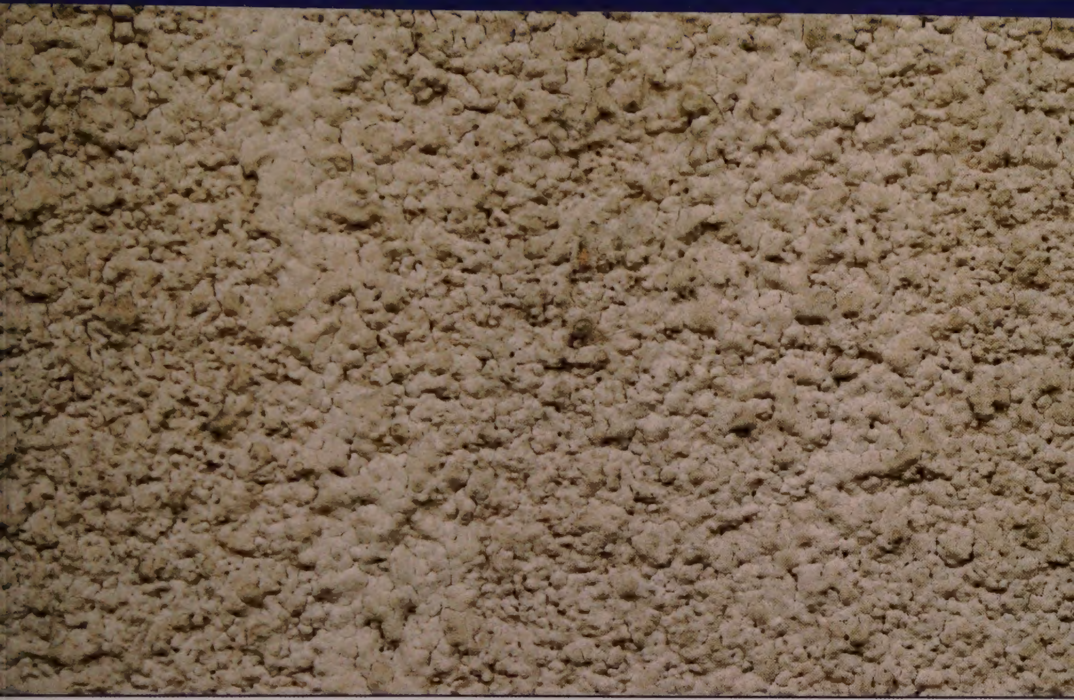

Parveen Jangra

Design & Development of Geopolymer Concrete

A detailed procedure for mix design of geopolymer concrete

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"Development and Design of Geopolymer Concrete"

I dedicate this thesis to
my family and beloved ones
for their constant support and unconditional love

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After an intensive period of four years, today is the day: writing this note of thanks is the finishing touch on my thesis. It has been a period of intense learning for me, not only in the scientific arena, but also on a personal level. Pursuing a Ph.D. thesis is a both painful and enjoyable experience. It's just like climbing a high peak, step by step, accompanied with bitterness, hardships, frustration, encouragement and trust and with so many people's kind help. When I found myself at the top enjoying the beautiful scenery, I realized that it was, in fact, teamwork and encouragement of numerous people including my well-wishers, my friends, colleagues and various institutions that got me there. Though it will not be enough to express my gratitude in words to all those people who helped me, I would like to reflect on the people who have contributed in many ways to the success of this study and made it an unforgettable experience for me.

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ABSTRACT

Concrete is among one of the most energy-intensive construction materials and has an immense impact on the environment as Ordinary Portland Cement (OPC), the main ingredient of the concrete being used as a binder in the matrix. Concrete used in construction at present also provides an approach to be environmental friendly construction material today as it consumes industrial wastes such as fly ash (FA), metakaolin, rice husk ash (RHA), granulated blast furnace slag (GBBS), etc. However, OPC contrary to this fact is responsible for the emission of carbon di-oxide (CO_2) which is the main greenhouse gas causing global warming and world's 5-7% CO_2 is caused by cement industry itself. The need to reduce the environmental pollution associated with the production of OPC and utilization of industrial wastes as a binder gained its popularity in the recent years to develop alternative construction material as binders in concrete.

Geopolymer concrete has emerged as a novel construction material and is one of those technologies where research is attempting for the last two decades to replace with OPC. Also, it has been established that geopolymer can act as an alternative binder to OPC in the concrete. Apart from its several technical advantages like good physical, chemical and mechanical properties geopolymer concrete adds sustainability to the environment as it has significantly smaller greenhouse footprint than Portland cement binders. Geopolymer binders are a class of alumino-silicates based materials. Geopolymers are synthesized by thermal activation of solid alumino-silicate materials such as fly ash (FA), metakaolin, rice husk ash (RHA), granulated blast furnace slag (GGBS), etc., with an alkali metal hydroxide and silicate solution.

The literature available on geopolymer concrete confirms that previous research was focused on the properties of low calcium fly ash based geopolymer concrete through heat curing. Literature is scanty on the slag and rice husk ash based geopolymer concrete. However, a few studies available reveal that geopolymer concrete can be produced at ambient curing by partially replacing the fly ash with other alternative materials. Further, studies are needed to investigate the properties of the geopolymer concrete by using other different waste materials like, RHA and GGBS which are rich in aluminous and silicates. A rational approach for the mix design of the geopolymer concrete targeting the required workability and compressive strength is not available. Available literature so far provides different hit and trial approaches wherein researchers chose random mixes and achieved required strength through heat curing

only. Such attempts have restricted the potential of this novel material. There should have been a mix design procedure which can target the required workability and compressive strength of the geopolymer concrete cured at ambient as well as high temperature and satisfy the need of the construction industry. Therefore, the present study has been carried out with the following objectives by using FA, RHA and GGBS at different curing temperatures including the ambient curing.

Objectives:

1. To develop GPC mix design method using FA, RHA and GGBS at ambient curing (at room temperature 27°C).
2. To develop GPC mix design method using FA, RHA and GGBS at heat curing (60°C & 90°C).
3. To study the structural properties of the GPC like compressive, split tensile and flexural strengths by varying the binder content, temperature and at different concentration of alkaline solution.
4. To validate the developed design mix method for its practical applications on large scale into the construction industry.

All the ingredients of the geopolymer concrete like fine and coarse aggregates, industrial waste materials (FA, RHA & GGBS) and alkaline activators (sodium hydroxide and sodium silicate) were collected locally which satisfied the requirements of relevant Indian standards. In this study, Alccofine 1203 (AF), was also used as an additive to improve the properties of fresh and hardened concrete. Further, a Naphthalene Sulphonate based superplasticiser confirming to the requirements of IS 9103:1999 was also included to improve the workability of fresh geopolymer concrete. A preliminary study was carried out for the selection of a mix proportion and important variables from the ingredients like alccofine content, silicate to hydroxide, fly ash to alkaline activated (AAL) and water to geopolymer binder (GPB) ratios and mix proportions to develop mix design method as detailed in the objectives.

To study the effects of alccofine in the geopolymer matrix, it was added with 0, 5 and 10% of the binder material by weight during the preliminary studies. It was decided that 10% alccofine can fulfil the requirements of the objectives. The literature revealed that both properties and manufacturing cost are affected by silicate to hydroxide ratio. As silicate is cheaper than hydroxide, it was decided that silicate to hydroxide ratio should be kept 2.5 in order to achieve the required strength and workability by keeping in mind the economy. In order to target the required compressive strength, geopolymer concrete was prepared at ambient (27°C) and at heat curing. Further, heat curing was done at 60°C and 90°C. As stated earlier, the quantum of alumino-silicate based materials affects the fresh and hardened properties of the GPC, samples were prepared by varying their quantities (350, 375 and 400 kg/cum. Sodium hydroxide with 8M, 12M and 16M concentrations was used to achieve the maximum slump and compressive strength as the properties of geopolymer concrete during fresh and hardened state have significant effect of its concentration. Binder (waste materials such as FA, RHA and GGBS) to alkaline activator solution ratio was kept 0.45 to meet the workability demands. To measure the rate of gaining the strength of the GPC samples, testing was done after 3, 7 and 28 days using Indian standard methods.

This study has been divided into two parts. First part presents the fresh and hardened properties of the geopolymer concrete produced with different NaOH concentration, quantities of waste material and curing temperatures. During the preliminary study, geopolymer concrete was developed using unprocessed fly ash and without alccofine and, thereafter, alccofine and processed fly ash were incorporated into the matrix. The workability of the fresh geopolymer concrete was measured in terms of slump and compaction factor. It was observed that the workability of geopolymer concrete prepared using unprocessed fly ash was very low and it might have been due to more carbon content, hygroscopic nature, high viscosity of alkaline solution which the mixture of sodium silicate and sodium hydroxide had, and fineness of the binder material. Further, observed compressive strength of the unprocessed fly ash based GPC (14MPa at heat curing) was not in the range of satisfying the minimum criteria of the concrete mix which is required for regular construction works. Therefore, unprocessed fly ash was replaced with processed fly ash and 10% of the alccofine was fixed for further research work. Also, a Naphthalene Sulphonate based water reducing superplasticizer (2% of the binder material i.e FA, RHA and GGBS) was used in all the mixes to improve the workability. This resulted in remarkable betterment in workability with the slump values from 50 to 160mm, 40 to 145mm and 40 to 140mm for FA, RHA and GGBS based geopolymer concrete, respectively.

It was observed that workability increased with the increase in quantum of waste material and in the presence of Naphthalene Sulphonate. Fineness of the alumino-silicate materials affected the slump values and the same was noticed with different type of binders. However, increasing trend in the slump values was observed when binder content was increased from

350 kg/cum to 400 kg/cum and trend was similar for all the three binders (FA, RHA and GGBS).

Workability decreased when concentration of the sodium hydroxide was increased. Further, the decrease in the slump values with the increase in NaOH molarity was due to the high viscosity of the alkaline solution as the available water decreased. Also, presence of lime in the alccofine and waste materials generates heat as a result of hydration which accelerates the hardening process of the GPC in fresh state and decreases the slump values. However, slump values were good enough to produce a workable mix and workability followed the same trend when measured through compaction factor.

It was observed that compressive, split and flexural strengths of the geopolymer concrete increase with the increase in concentration of the sodium hydroxide, curing temperature and higher amount of binder/waste materials used. The trend was similar for FA, RHA and GGBS based concrete in the presence of alccofine. A remarkable maximum compressive strength of 41MPa to 73MP, 39MPa to 71MPa and 57MPa to 82MPa was recorded for FA, RHA and GGBS based GPC, cured from ambiently to 90°C. Similarly, a maximum split tensile strength of 4.41 to 5.18MPa, 3.64 to 5.10MPa and 4.52 to 5.57MPa, also, flexural strength of 4.23 to 5.64MPa, 4.12 to 5.56MPa and 5.05 to 6.08MPa were recorded for FA, RHA and GGBS based GPC, respectively, when temperature increased from ambient (27°C) to 90°C.

Maximum strength in all the cases was recorded at 16M NaOH concentration and at 400 kg/cum of waste material in the form of FA, RHA and GGBS along with 10% of alccofine. When strength of all the GPC mixes prepared with different waste materials was compared, it was found that maximum strength was obtained with GGBS. Maximum strength in the case of GGBS was due to its maximum CaO content when compared with others.

When microstructure of the fly ash based geopolymer concrete was studied, it was found that presence of lime in both aluminous silicate materials and alccofine combined the polymerisation reaction with hydration. This resulted in the formation of silicate hydrates such as sodium aluminate silicate hydroxide (NASH) and calcium aluminate silicate hydroxide (CASH) which resulted into higher strength. Also, when additional water was added during mixing, lime reacted with water and binder resulting into the formation of CSH and confirmed the presence of hydration. The combination of hydration and polymeric reactions provided the required early strength even upto 44MPa at 28days at ambient temperature.

Alccofine which is rich in lime content, formed CSH on hydration when extra water was added and heat was developed into the matrix which further accelerated the polymeric reactions and that is why early required strength could be achieved even at ambient temperature.

Further, maximum strength in all the cases was recorded when GPC samples were cured at 90°C. The compressive strength of RHA(400kg/cum) based GPC sample which was prepared using 12M and cured at room temperature was found to be 36MPa and when temperature changed to 90°C the strength increased to 53MPa. It happened so as geopolymer mechanism involves polymeric reactions that accelerate with high temperature as such most of the strength was achieved at an early stage when heat curing was adopted instead of ambient temperature.

Compressive strength of ambiently cured GPC using 375kg/cum fly ash as binder and 12M hydroxide solution was recorded as 10MPa, 17MPa and 35MPa at 3, 7 and 28days, respectively. Further, when FA was replaced with RHA keeping the other ingredients same, the change in compressive strength recorded was 8MPa, 15Mpa and 35MPa. Therefore, it can be concluded that rate of increase in strength with age in the case of FA and RHA based GPC cured at 27°C is similar to that of conventional concrete which has already been studied by various researchers.

Based upon the results of compressive strength, design aids (AF-GPC graphs) were developed considering all the suitable variables such concentration of the NaOH, binders quantum, temperature etc. which influenced the properties of the geopolymer concrete.

Second part of this study is basically the analysis of the results and it presents the procedure for mix design of the geopolymer concrete along with its validation. Graphs were developed using the compressive strength results. These graphs covered the variation of compressive strength with the important variables like quantity of raw materials (FA, RHA and GGBS), NaOH molarity and different temperature range. Slump values were used to develop the workability bands as per the placing conditions and using the clause 7 of IS 456:2000. The essential features of the proposed method are the flexibility to select binder content, molarity of the sodium hydroxide and curing temperature required for specific strength and workability. Further, graphs have been proposed for 3, 7 and 28 days compressive strength. The rational mix design approach was validated with the help of the examples for all three binders by considering the ambient and heat curing. Geopolymer concrete of grade ranging from 10MPa to 70MPa can be designed using the design aids. To check the proposed methodology, two mix designs targeting the compressive strength and workability, for each binder material were designed. TM25 and TM35 for FA, TM15 and TM25 for RHA and TM35 and TM50 for GGBS based GPC were designed (where, TM-50: trial mix- target strength in MPa). Target slump for FA and RHA based GPC was 75mm and for GGBS based GPC it was 100mm. Different range of the mixes were selected to cover a wide range over different parameters of geopolymer concrete. It was found that the proposed methodology gave consistent results. Proposed methodology was found to be valid for all three binders i.e. FA, RHA and GGBS. Further, the proposed methodology can also be used to develop the geopolymer concrete at ambient temperature. It was concluded that by strictly following the proposed procedure, required geopolymer concrete with targeted compressive strength and slump can be produced effectively and efficiently. Moreover, the consistent results obtained in the validation procedure show the ability of the method to serve the industrial and commercial demands.

To cover the gap in the available literature in the field structural design of GPC, stress-strain curves were developed by testing the cylinders of 150mm x 300mm size in addition to cubes (150mm size) and beams (100mm x 100mm x 500mm). It was noticed that strain at the peak stress was in the range of 0.0025 to 0.0038. Also, strain at failure was in the range of 0.0035 to 0.006. Young's modulus of elasticity ranged from 0.215 x 10^5 MPa to 0.381 x 10^5 MPa and 0.157 x 10^5 MPa to 0.313 x 10^5 MPa for heat and ambient cured geopolymer concrete specimens, respectively. Further, both these ranges were similar for FA, RHA and GGBS based GPC specimens. Both these measured values were very much similar to the values obtained in the case of conventional concrete by various researchers. Therefore, results of this study confirmed that GPC had the same behaviour that obtained in the case of conventional concrete. Hence, same design methodologies that is available for conventional concrete can be used to design the geopolymer concrete structural members.

It can be concluded from the results of this study that geopolymer concrete by using FA, RHA and GGBS can be developed at ambient as well as at heat curing with the inclusion of alccofine for the required compressive strength. A mix design method to produce GPC targeting the compressive strength and workability at the same time, for all three binders has been suggested and validated. This study as such extends the applicability of geopolymer concrete beyond precast concrete industry to general purpose. Further, results of these study show that GPC and OPC based concrete has similar behaviour so, structural design methodologies for both the concretes would be same.

ABBREVIATIONS

FA	Fly Ash
RHA	Rice Husk Ash
GGBS	Ground Granulated Blast-Furnace Slag
C-S-H	Calcium Silicate Hydrate
NASH	Sodium Aluminate Silicate Hydrate
CASH	Calcium Aluminate Silicate Hydrate
AS	Alkali Activated Slag
OPC	Ordinary Portland Cement
w/GPB	Water to Geopolymer Binder Ratio
w/GPS	Water to Geopolymer Solid Ratio
SSD	Saturated Surface Dry
CTL	Control
GHG	Greenhouse Gases
LCA	Life cycle analysis
ASR	Alkali silica reaction
CKD	Cement Kiln Dust
GP	Geopolymer
GPC	Geopolymer Concrete
AAL	Alkaline Activator Solution
ASH	Alumino Silicate Hydrate
AL/W	Alkaline Liquid to Water Ratio
AL/FA	Alkaline Liquid to Fly Ash Ratio
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
XRF	X-ray Fluorescence Spectroscopy
RMC	Ready Mix Concrete
PSD	Particle Size Distribution
AF	Alccofine
SS	Sodium Silicate
SH	Sodium Hydroxide
ST	Slump Test
CF	Compaction Factor

CS	Compressive Strength
FS	Flexural Strength
STS	Split Tensile Strength
WOH	Weight of Water Present in Hydroxide Solution
WSI	Weight of Water Present in Silicate Solution
SP	Superplasticizer
MPa	Megapascal
Cum	Cubic Meter
MM	Millimetre
M	Molarity

CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER - 1

INTRODUCTION

Concrete, except water, concrete is the most widely used building material in the world. The production of the integral constituent, ordinary Portland cement (OPC), proves to be unsustainable with regards to it's the environmental impact and it involves energy intensive process and emissions of carbon dioxide (CO_2). However, the requirement for cement and concrete will be substantial for a stronger and durable construction material, until an equally effective and economic alternative is available. Therefore, it is necessary to pay attention on severe environmental impact of standard concrete production. Concrete International recognizes the situation at hand, and the article titled "Sustainable Development and Concrete Technology" quotes the current issues. 'The contribution of OPC worldwide to greenhouse gas emissions is estimated to be approximately 1.35 billion tons annually or approximately 7% of the total greenhouse gas (GHG) emissions to the earth's atmosphere [1]. The reason large amounts of CO_2 are released during the manufacturing of cement is due to immense heat involved in the process. The clinker (primary material to produce the cement) is produced at temperatures of 1400°C, and therefore, energy requirements to yield this temperature account for approximately half of the released CO_2 in the production of cement, the second half is released during the calcination process in which calcium carbonate is reduced to calcium oxide [2]. The production of cement alone accounts for approximately 5% of the worlds carbon dioxide emissions and further, G20 was responsible for 81.5% of global CO_2 emission in 2015 [3-6]. According to the International Energy Agency, approximately 0.81 kilograms of CO_2 is generated per kilogram of cement produced annually throughout the world. The production of cement also produces millions of tons of Cement Kiln Dust (CKD) which is harmful to the respiratory system [7]. Further, the continuing release of GHG through the burning of fossil fuels and dumping of the waste materials into ground, increased the risk of a rise in average surface temperatures and it have a negative effect on the sea levels. Therefore, it is need of the hour to develop an environmentally friendly construction material [8]. In recent times, researchers have attempted to produce concrete as an environmentally friendly product by replacing amounts of ordinary Portland cement from the mix with industrial by-products such as fly ash (FA), rice husk ash (RHA) and ground granulated blast furnace slag (GGBS). Huntzinger et al. (2009) [9] use life-cycle analysis (LCA) to evaluate the environmental impacts and, therefore, the 'global warming factor' of the manufacture of Portland cement. Substituting natural pozzolans for OPC will effectively reduce the 'global warming factor' of the product proportional to the amount replaced [9].

It is ample clear from the above that most of the previous research available highlights the current situation of cement production and the damage caused to the environment. The next logical step into this investigation is to either prevent this damage or offer alternatives binder to OPC to produce concrete. It is in the opinion of many researchers, that the use of ordinary Portland cement in concrete is not going to slow down, despite the ongoing research into alternative binders. But results obtained from the research of last three decades have proven this hypothesis wrong.

It would, therefore, be a sustainable decision to investigate the alternative binder material to produce the concrete. Further, the alternative material should have inherent advantages like relatively low cost of production, ease of handling, capacity to be moulded into desired shape, achievement of desired strength ranging from low to very high, serviceability and durability. Moreover, it should replace the binder of the concrete and which have less footprints of greenhouse gases.

1.1 ALTERNATE MATERIAL

It is therefore ample obvious from the above discussions that the construction material in future should be easily produced, durable, strong and most important environment friendly. The pollution effects on environment can be reduced by increasing the usage of industrial by-products in our construction industry. By product materials such as fly ash, granulated blast furnace slag, silica fume and rice-husk ash is an environmental threat to the public, if not disposed properly. Deposition of these materials in storage places can have a negative influence on water and soil because of its granulometric and mineral composition as well as morphology and filtration properties [10]. Therefore, the safe disposal of these materials is still a major concern. Researchers have tried the partial replacement of cement with waste materials which possess cementitious properties [11-13]. However, partial replacement of cement with supplementary materials in concrete reduces the release of CO_2 gas only to a limited extent, and a complete replacement is always preferable.

One possible alternative is the use of alkali-activated binder using industrial by-products containing silicate materials [14]. The most common industrial by-products used as binder materials are fly ash (FA), Rice husk ash (RHA) and ground granulated blast furnace slag (GGBS). These waste materials are rich in silica and CaO content which when activated using alkaline solutions resulted into a good binder to produce hard material as conventional concrete.

1.2 MECHANISM

Recent research has shown that it is possible to use 100% waste as the binder in concrete by activating them with an alkali component, such as; caustic alkalis, silicate salts, and non-silicate salts of weak acids [15, 16]. There are two models of alkali activation. Activation by low to mild alkali of a material containing primarily silicate and calcium will produce calcium silicate hydrate gel (C-S-H), similar to that formed in Portland cements, but with a lower Ca / Si ratio [17, 18]. The second mechanism involves the activation of material containing primarily silicate and aluminates using a highly alkaline solution. These binders (FA, RHA & GGBS) when activated with alkaline solution, reactions take place which lead to the formation of an inorganic binder through a polymerization process [19, 20]. The alkali used as the activator tends to be an alkali silicate solution such as sodium silicate (water glass) but can also be sodium hydroxide solution, or a combination of the two, or other source of alkali (such as lime). The term "Geopolymeric" is used to characterise this type of reaction and accordingly, the name geopolymer has been adopted for this type of binder [21]. The geopolymeric reaction differentiates geopolymer from other types of alkali activated materials (such as; alkali activated slag) since the product is a polymer rather than C-S-H gel. The new class of material developed using alkali activators, waste materials, aggregates and water is termed as "Geopolymer concrete". The term ''geopolymers'' was first introduced to the world by Davidovits of France resulting in a new field of research and technology. Davidovits explained that geosynthesis is the science of manufacturing artificial rock at a temperature below 100°C in order to obtain natural characteristics (hardness, longevity and heat stability) of rock. Geopolymers can be thus viewed as mineral polymers resulting from geochemistry or geosynthesis.

1.3 GEOPOLYMER CONCRETE

Geopolymer is a new class of construction material which have geopolymer (GP) as the main binder to bind the other ingredients of the system like fine and coarse aggregates. There are two main ingredients required for development of geopolymer binders:

- Geopolymeric source materials (GSMs) rich in silica and alumina, which could be natural minerals (such as kaolinite, clays, etc) or industrial by-products (such as fly ash, silica fume, slag, rice-husk ash etc).

- Alkaline Activator Solution (AAS) based on alkali metals (commonly Sodium or Potassium) based. The most common AAS is a combination of alkali hydroxide (NaOH, KOH) and alkali silicate (Sodium or potassium silicate).

Geopolymers made from calcined source materials, such as metakaolin (calcined kaolin), fly ash, slag etc., yield higher compressive strength when compared to those synthesised from non-calcined materials, such as kaolin clay. The source material used for geopolymerisation can be a single material such as fly ash, slag etc. or a combination of several types of materials [22, 23].

Le et al. (2009) studied the mechanical properties of basalt fibre reinforced geopolymeric concrete (BFRGC), including dynamic compressive strength, deformation and energy absorption capacity [24]. Tianyu et al. (2014) did an experimental studies on the behaviour of low calcium and bottom ash-based GPC cured at ambient temperature [25]. Vijai et al. (2012) studied the properties of glass fibre geopolymer concrete [26]. Joseph et al. (2012) studied the influence of aggregate content on the behaviour of fly ash based GPC [27]. He et al. (2013) developed a new type of geopolymer composite, synthesized from two industrial wastes, red mud (RM) and rice husk ash (RHA), at varying mixing ratios of raw materials [28]. Adak et al. (2014) in their research discussed the effect of nano-silica on the strength and durability of fly ash based geopolymer mortar to get the rid from heat curing [29]. A research on the effects of fly ash fineness, nano silica, and curing types on mechanical and durability properties of fly ash mortars was carried out by Hasan et al. (2014) [30]. Hardjito et al. (2015) developed the geopolymer concrete and studied the effects of various parameters on the strength [31].

Hardjito et al. (2005) in their study developed GPC by using conventional concrete practices and focussed to identify the salient parameters that influence the mixture proportions using low calcium fly ash-based geopolymer concrete [32]. Aleem et al. (2012) reported the optimum mix design in terms of fly ash: fine aggregate: coarse aggregate was 1:1.5:3.3 with a solution (NaOH & Na_2SiO_3 combined together) to fly ash ratio of 0.35 and maximum strength achieved was 48MPa [33]. Ramujee et al. (2013) identified the main governing factors in the geopolymer matrix and tried an attempt to develop the mix design method for geopolymer concrete for low, medium and higher grades by targeting the compressive strength [34]. Patankar et al. (2015) developed a systematic approach for fly ash based GPC but maximum grade that could be selected from this study was of M40. Junaid et al. (2015) attempted to develop design aids for fly ash based GPC but selection of fly ash content was totally based on hit and trail approach [35]. Pavithra et al. (2016) developed a mix design methodology for fly ash based GPC and compressive strengths ranging from 23 to 53 MPa at varying activator solution to fly ash ratio was achieved [36]. Again, provision for targeting the workability was not mentioned.

Hardjito et al. (2004) studied the behaviour of the geopolymer concrete by using stress strain behaviour and it was concluded from their study that GPC has similar behaviour that obtained for conventional concrete[37]. Noushini et al. (2016) developed the model for heat cured GPC and compared with the already generated models for stress strain relationship, it was concluded from their study that the proposed relationship was very much similar to the relationship proposed by Hardjito [38]. Junaid et al. (2016) studied the stress-strain behaviour of fly ash based geopolymer concrete at elevated temperatures (upto 800°C) and brittle failure was obtained for all the specimens with strains at peak stress were in the range of 0.00337 mm/mm (minimum) to 0.006159 mm/mm (maximum) [39].

Although, numerous studies on low calcium fly ash based geopolymer concrete at heat curing have been carried out worldwide, the majority have focused on material characterisation, the enhancement of physical and chemical properties of the material, the effects of source material and engineering properties [11, 12, 40-42]. There has not been an extended study which gives idea to develop a mix of desired strength and workability. It is felt that this sustainable material will have wider acceptability if mix design method targeting compressive strength and workability can be developed, required strength can be achieved at ambient temperature and design tools are made available. Also, information regarding the structural design of the geopolymer concrete members is up to a limited extent. The present research is therefore dedicated to develop the environment friendly geopolymer concrete in Indian context, to formulate a mix design approach for the geopolymer concrete cured at ambient as well as elevated heat and to check whether the available structural design methodologies of the conventional concrete members are applicable to geopolymer concrete structural members or not.

1.4 OBJECTIVES OF THE INVESTIGATION

Geopolymer concrete not only reduces the CO_2 by utilizing industrial by products but it also proves to be an efficient substitute to ordinary Portland cement (OPC). The aim of the research is to study the behaviour of FA, RHA and GGBS based geopolymer as an alternative to the use of ordinary Portland cement (OPC) in the production of concrete. The distinct goals will include;

1. To develop the GPC using different waste products in Indian context with the focus on achieving minimum required compressive strength as per Indian standards.
2. To develop GPC mix design method using FA, RHA and GGBS at ambient curing (at room temperature 27°C).
3. To develop GPC mix design method using FA, RHA and GGBS at heat curing (60°C & 90°C).
4. To study the structural properties of the GPC like compressive, split tensile and flexural strengths by varying the binder content, temperature and at different concentration of alkaline solution.
5. To validate the developed design mix method for its practical applications on large scale into the construction industry.

1.5 ORGANISATION OF THE THESIS

This report is structured as follows; **Chapter one** describes the motivation for developing FA, RHA and GGBS based geopolymer as an alternative binder for concrete.

Chapter two reviews the literature related to the environmental tribulations of ordinary Portland cement, the alternatives to mixing concrete utilizing OPC, the reaction mechanism, properties and the previous research conducted in the use of FA, RHA and GGBS based geopolymer concrete. The general background of geopolymer concrete production is investigated, along with mix proportioning, mixing procedures and curing properties.

Chapter three describes the experimental process in conducting the research for this report. Attention is paid to the materials used, mix designs, mixing procedures, curing conditions and the method of testing the geopolymer concrete specimens produced etc.

Chapter four reports on the experimental results obtained and discussion on the properties of the GPC.

Chapter five reports on analysis of the results, the mix design development of RHA, GGFBS and fly ash based geopolymer concrete.

The present report's conclusions and recommendations are included in **Chapter six**. This section is based upon all results and observations discovered in the research throughout the years. Further to this, a list of references & list of publications is given in the end.

CHAPTER – 2

LITERATURE REVIEW

2.1 INTRODUCTION

This chapter presents details of recent research on geopolymers and geopolymer concrete, with an emphasis on geopolymer concrete prepared with fly ash, rice husk ash and ground granulated blast furnace slag. Review has done on the mix design, structural and durability properties. The important parameters effecting the properties of geopolymer concrete such as the source materials, curing temperature and period, Si/Al ratio in the mix, alkali concentration, etc., have been reviewed and discussed here to identify the objectives of the study.

2.2 HISTORY OF THE GEOPOLYMER

The method of making cementitious materials was known to human civilization even in the 8th century B.C. According to Davidovits, the technique of making a sort of cement paste by dissolution of rocks and using this paste to agglomerate aggregates or/and sands was used during this period for making statues and large stone blocks [43]. Also, the large stone blocks used to construct the pyramid of the Pharaoh at Cheops were cast in place with this technique [44]. However, this hypothesis has not been accepted fully by many [45]. Ancient terra-cotta vases of the 7th to 9th century was made of earth and by using a method of low temperature synthesis (up to 200 °C) on the mixture of clay soils and alkalis.

Prudon also carried out investigation on the formation of alkali activated cement (binder) in 1940 [46]. The investigator used blast furnace slag as alumino-silicate material and sodium hydroxide as alkali. Since then, alkali activation studies were carried out in different countries but it picked up momentum only in the 1990s. Roy [47] compiled the history of the development of alkali-activated cement and the same is reproduced in Table 2.1.

Table 2. 1: History of alkali activated binders

Sl.No	Year	Author	Significance
1	1939	Feret	Slag used for cement
2	1940	Purdon	Alkali- slag combinations
3	1959	Glukhovsky	Theoretical basis and development of alkaline Cement
4	1965	Glukhovsky	First called "alkaline cement"
5	1979	Davidovits	"Geopolymer" term indroduced

6	1979	Malinowsky	Ancient aqueducts characterized.
7	1983	Forss	Clinger free cement (slag-alkalisuperplsticizer)
8	1984	Langton and Roy	Ancient building materials characterized
9	1985	Davidovits	Patent of “Pyrament” cement
10	1986	Krivenko	DSc thesis, R_2O- Al_2O_3-SiO_2- H_2O
11	1986	Malolepsy and Petri	Activation of synthetic melilite slags
12	1986	Malek. et al.	Slag cement-low level radioactive wastes forms
13	1987	Davidovits	Ancient and modern concretes compared
14	1989	Deja and Malolepsy	Resistance to chlorides shown
15	1989	Kaushal et al.	Adiabatic cured nuclear wastes forms from alkaline mixtures
16	1989	Roy and Langton	Ancient concretes analogs
17	1989	Majundar et al.	Monocalcium Aluminate – slag activation
18	1989	Talling and Brandstetr	Alkali-activated slag
19	1990	Wu et al.	Activation of slag cement
20	1991	Roy et al.	Rapid setting alkali-activated cements
21	1992	Roy and Silsbee	Alkali-activated cements: an overview
22	1992	Palomo and Glasser	CBC (Chemically bonded cement) with Metakaolin
23	1993	Roy and Malek	Slag cement
24	1994	Glukhovsky	Ancient, modern and future concretes 25 Krivenko
25	1994	Krivenko	Alkaline cements
26	1995	Wang and Scrivener	Slag and alkali-activated microstructure

Different terminologies had been used by investigators for the products developed using geopolymerization synthesis since 1940s like “soil silicate concrete", "soil cement”, “alkali - activated cement”, “inorganic cement” etc. [48, 49]. However, the most widely accepted terminology was the term “geopolymer”, as coined by Davidovits in 1979 [50]. Davidovit selected the name “Geopolymer” because of the similarities with organic condensation of polymers as far as their hydro thermal synthesis conditions are concerned.

2.3 CHEMISTRY OF GEOPOLYMER CONCRETE

In GPC, alumina (Al_2O_3) and silica (SiO_2) rich compounds react with metal alkalis to form binders. Such binders provide bond to coarse and fine aggregates and form concrete, usually known as geopolymer concrete (GPC). The source of the alumino-silicates can be fly ash, silica fume, rice husk ash and/or slag [51, 52]. As these alumino-silicate rich materials are industrial by-products and, in contrast with OPC, do not require calcination, replacing each cubic metre of OPC with GPC can reduce emissions of CO_2 between 45% and 80% [53, 54].

The chemical composition of the geopolymer material is similar to natural zeolitic materials, but the microstructure is amorphous [55]. The polymerization process involves a substantially fast chemical reaction under alkaline conditions on Si-Al minerals, resulting in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds [56]. The formed gel product contains alkaline cations which compensate for the deficit charges associated with the aluminum-for-silicon substitution [57]. An intermediate, aluminium-rich phase is first formed which gives way to a more stable, silicon rich three-dimensional gel product of form $Q_4(nAl)$, further dependent upon curing conditions and activator type [58].

Weng and Sagoe-Crentsil [59] presented the chemistry associated with the formation of geopolymer system having low Si-Al ratio, generally referred to as poly (sialate) geopolymer system. They also proposed three steps during the formation of geopolymer namely dissolution, hydrolysis and condensation. They represented the dissolution and hydrolysis process as follows.

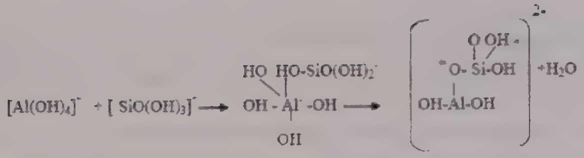
$$Al_2O_3 + 3H_2O + 2OH^- \longrightarrow 2Al(OH)_4^-$$

$$SiO_2 + H_2O + OH^- \longrightarrow [SiO(OH)_3]^-$$

$$SiO_2 + 2OH^- \longrightarrow [SiO_2(OH)_2]^{2-}$$

These reactions established that H_2O molecules and OH^- iron were consumed with continuous dissolution.

During the condensation reaction between $Al(OH)_4^-$ and $[SiO(OH)_3]^-$, the $[Al(OH)_4]^-$ and $[SiO(OH)_3]^-$ species are linked to each other by the attraction between one of the OH groups from $[SiO(OH)_3]^-$ and Al ion of $[Al(OH)_4]^-$, which results in an intermediate complex (Alumino-silicate hydrates). The two OH group in the intermediate complex then condense to form an alumino-silicate species by releasing H_2O molecules. The following equation explains this condensation reaction.



However, the most widely accepted mechanism consists of three reaction stages namely dissolution, hydrolysis and polycondensation [59-62].

Water is not involved in the chemical reaction of geopolymer concrete and instead water is expelled during curing and subsequent drying. This was in contrast to the hydration reactions that occur when Portland cement was mixed with water. Such differences had a significant impact on the mechanical and chemical properties of the resulting geopolymer concrete, and also renders it more resistant to heat, water ingress, alkali–aggregate reactivity, and other types of chemical attack [63, 64].

Although details are still debated, many researchers agree that the basic geopolymer reaction mechanism is in three stages; namely: dissolution of Si and Al from the source material, hydrolysis or gelation, and condensation forming a 3D network of silico-aluminates also termed as the 'geopolymer backbone' [52, 65-68]. Earlier, fly ash has been used in GPC as a source of alumina (Al_2O_3) and silica (SiO_2). However, it is very clear that the role of fly-ash in GPC is entirely different from that it plays when used as a cement replacement material in conventional concrete to enhance certain properties such as workability or to reduce the heat of hydration [69]. In such cases fly-ash has no pronounced effect on the strength of concrete [69] especially early strength. In GPC, however, fly ash is the sole source of alumino-silicates which react with the alkaline medium to form the geopolymer backbone and is thus responsible for the GPC's strength. Fly ash, however, does come in different classifications according to its composition. For instance, Class C fly ash, which is relatively rich in CaO, tends to set far too quick to be used in any practical application [65]. Additionally, the high amounts of CaO may lead to durability issues in GPC made from Class C fly ash [70, 71]. However, recently study based on the low calcium fly ash incorporating alccofine in the geopolymer concrete resulted into improved durability in terms of permeability [72]. Owing to the above results, the focus here is on low calcium (Class F) fly ash, rice husk ash and ground granulated blast furnace slag based alkaline activated geopolymer concrete systems.

2.4 PRODUCTION OF GEOPOLYMER CONCRETE

2.4.1 Ingredients of the geopolymer concrete

There are two types of materials which are required to make geopolymer concrete. One is the source material containing alumina and silica and other is an alkali solution that activates the polymerization reaction.

2.4.1.1 Source materials

The source materials (alumino-silicate) may be natural minerals, such as kaolinite, calcined kaolinite (metakaolin) and clays [23, 24, 30, 31]. The source material should be amorphous and degree of polymerization mainly depends on the degree of amorphosity and fineness of alumino-silicate materials. Kaolinite is a clay mineral having the chemical composition $Al_2Si_2O_5(OH)_4$. Rocks that are rich in kaolinite are known as kaolin or china clay. Metakaolin is manufactured by the dehydration of kaolinite at temperature ranging between 550°C to 900°C. Other clay minerals containing oxides of alumina and silica are also used as source material for making geopolymer [40].

Alternatively, industry waste products such as fly ash, slag, red mud, rice-husk ash and silica fume may be used as source material for the synthesis of geopolymers [32-39]. Fly ash is an industrial waste produced in Thermal power stations where coal is used as the fuel. Slag is formed in blast furnace during the manufacturing process of iron from its ore. Red mud is an industrial waste produced in Aluminium manufacturing industry where Bauxite is used as the raw material. Rice Husk is produced by the controlled burning of raw rice husk. Silica fume is a by-product of producing silicon metal or ferrosilicon alloys. All these source materials are rich in alumina and silica [71].

2.4.1.2 Alkali activators

The alkali component used as an activator is a compound from the elements of the first group in the periodic table. The common activators are NaOH, Na_2SO_4, water glass, Na_2CO_3, K_2CO_3, KOH, K_2SO_4 or a little amount of cement clinker and complex alkali component [41]. For the preparation of the alkali solution a single alkali type or a mixture of different alkalis can be used. The most commonly used alkali for the manufacture of geopolymer is a mixture of the solutions of NaOH or KOH and Na_2SiO_3 [42, 43].

2.4.1.3 Mixing time

Mixing time significantly affects the fresh and hardened properties of the geopolymer concrete. Hardjito studied the effects of mixing time on the properties of GPC and concluded that with the increase in the mixing time slump values and strength decreased [31].

2.4.2 Factors affecting geopolymer concrete

The properties of constituent materials and the chemical composition in the geopolymers dominate the mechanical properties of the geopolymer end products, the same way for the conventional concrete. The geopolymerization reaction is very sensitive to different raw materials (particle size and distribution, crystallization degree, etc.), different alkali-activators (Sodium / potassium hydroxide, Sodium / potassium silicate, and the ratio of these two, etc.), different Si / Al ratios, different water / ash ratios, different curing conditions (temperature, moisture degree, opening or healing condition, curing time, etc.) [37, 73]. Different mechanical and thermal properties of geopolymer cement will be produced according to different raw materials, alkali-activators, Si-Al ratios, water-ash ratios, and curing conditions.

2.4.2.1 Curing temperature

The influence of curing temperature and curing time on the compressive strength of fly ash based-geopolymer paste had been studied by Palomo et al. (1999) [74], Swanepoel et al. (2002) [75] and van Jaarsveld et al. (2002) [76]. They found that both curing temperature and curing time influenced compressive strength. Compressive strength up to 60MPa was obtained when cured at 85°C for five hours. In addition, the utilisation of sodium hydroxide (NaOH) combined with sodium silicate solutions ($Na_2O.SiO_2$) resulted in the highest strength for the paste (Palomo et al., 1999) [74]. Swanepoel et al. (2002) [75] found that the optimum condition being curing was at 60°C for a period of 48 hours.

Van Jaarsveld et al. (2002) [76] also confirmed the importance of curing at an elevated temperature for fly ash-based geopolymer materials and observed that curing for a longer period of time at an elevated temperature weakened the microstructure and, therefore, reduces the compressive strength of fly ash-based geopolymer materials.

Bakharev (2005) [77, 78] investigated the influence of elevated temperature curing on phase composition, microstructure and strength development in ASTM Class F fly ash-based geopolymer materials with sodium silicate and sodium hydroxide solutions as alkaline activators. He found that long pre-curing at room temperature was beneficial for strength development as it allowed for shortening the time of heat treatment to achieve high strength. Samples with sodium silicate solution as activator were found to have more strength development in 6 hours of heat curing than 24 hours of standard curing at room temperature. An increase in curing temperature caused a decrease of Si / Al ratios in alumino-silicate gel which further resulted into less compressive strength, while long curing at room temperature narrowed the range in Si / Al ratios distribution.

Palomo et al. (1999) [74] studied curing of alkali activated fly ash (0.25 and 0.30 liquid / solid ratio) at 65°C and 85°C. They indicated that the compressive strength of geopolymers (8–12 M) cured at 85°C for 24 h was much higher than those cured at 65°C. However, the rise of strength was much smaller when curing time was extended after 24 h.

Perera et al. (2007) [79] studied the curing of metakaolin-based geopolymers under ambient (21–23 °C) and heat conditions (40– 60°C) with a controlled relative humidity (RH) for 24 h and found that curing at 30% RH was preferable than that at 70% RH.

Heah et al. (2011) concluded that the curing of metakaolin-based geopolymers at ambient temperature was not feasible while increase in temperature (40°C, 60°C, 80°C, 100°C) favoured the strength gain after 1-3 days. However, curing at higher temperature for a longer period of time caused failure of samples at a later age due to the thermolysis of –Si–O–Al–O– bond [80].

Rovnanik (2010) [81] reported that curing of metakaolin based geopolymer at elevated temperature (40–80°C) accelerated the strength development but in 28 days, the mechanical properties such as compressive strength deteriorated when compared with results obtained for an ambient or slightly decreased temperature.

Ebrahim and Ali (2009) [82] prepared three mixes with different formulations and cured hydrothermally at different temperatures (45, 65, 85°C) and time (5–20 h) after 1 and 7 days of pro-curing. Longer-procuring at room temperature, before the application of heat was beneficial for higher strength development [16]. In general, adequate curing of geopolymeric materials was required to achieve optimal mechanical and durability performance to maintain their structural integrity [76].

2.4.2.2 Admixtures

Kusbiantora et al. (2013) [83] reported from their studies that admixtures such as sucrose and citric acid which act as retarder in OPC had different mechanism in fly ash-based geopolymers. Sucrose acted as a retarder since it was absorbed by Ca, Al and Fe ions to form insoluble metal complexes. On the other hand, citric acid acted as an accelerator reducing the setting times by 9 and 16 min, respectively. Amongst the commercial superplasticizers, the naphthalene based superplasticizer was effective when single activator was used rendering 136% increase in relative slump without any decrease in compressive strength. Modified polycarboxylate based superplasticizer was efficient one when multi-compound activator was used with a decrease in compressive strength of 29% [84]. However, retarding effect of polycarboxylate based superplasticizer was also reported in fly ash / slag blended system though the improvement in workability was significant compared to naphthalene based superplasticizer [85].

2.4.2.3 Constituents effect

The most important factors affecting the properties of geopolymer pastes are: SiO_2-Al_2O_3 ratio, R_2O-Al_2O_3 ratio, SiO_2-R_2O ratio ($R = Na^+$ or K^+) and liquid–solid ratio. The majority of research concluded that an amorphous structure of geopolymers was preferable in order to achieve desired mechanical strength [22, 76, 86-89]. The relationship between the compressive strength and SiO_2-R_2O ratio showed that an increase in alkali content or decrease in silicate content increased the compressive strength of geopolymers attributable to the formation of alumino-silicate network structures [22, 76]. Geopolymer activated with NaOH alone with Si/Na of 4/4 or less formed the crystalline zeolite ($Na_{96}Al_{96}Sr_{96}O_{384}216H_2O$) but at a ratio >4/4, nanosized crystals of another zeolite ($Na_6[AlSiO_4]_6.4H_2O$) were formed [86]. The addition of even small amount of sodium silicate to the NaOH significantly reduced crystallite formation due to templating function of silicate units. At low activator dosage (18%), the pores developed in the fly ash-based paste were larger and exhibited wider distributions (19.8–2342 A) whereas at higher activator dosage (30%), the pores were smaller and showed a narrow distribution (19.8–1155 A) mainly due to the pore refinement as a result of more dissolution of particles and formation of reaction products (Fig. 2). The reduced porosity enhanced the strength of geopolymer pastes [87]. Typically, the optimum geopolymer strength was reported with SiO_2-Al_2O_3 ratio in the range of 3.0–3.8 and Na_2O-Al_2O_3 ratio of ~1 [88, 89]. Changes in SiO_2-Al_2O_3 ratio beyond this range had been found to result in low strength. The setting time of geopolymer pastes increased with increasing SiO_2-Al_2O_3 ratio of the initial mixture.

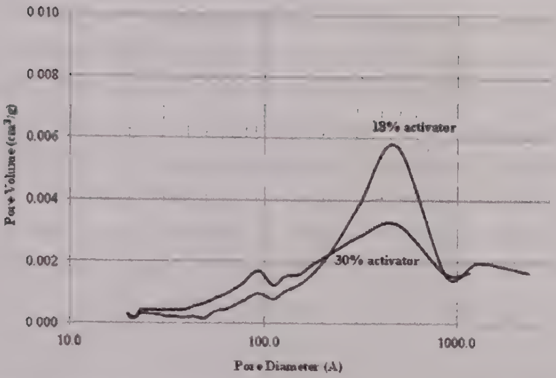


Fig. 2. 1: Pore size distribution of fly ash-based geopolymer pastes at different *activator dosages [87].*

2.4.2.4 C-S-H phase effect

The effect of C-S-H phase on the geopolymerization of alumino-silicates has been studied with a view to know its role in early age strength [12, 90-95]. In metakaoin and slag blends, both C-S-H phase and alumino-silicate gel (N-A-S-H) co-existed in the paste [90] as similar to NaOH activated high calcium fly-ash based geopolymer [91] which are responsible for the strength increase. When natural calcium silicate minerals were added at lower alkalinity, the little dissolution of calcium occurred which resulted in less C-S-H gel formation and subsequent strength reduction of geopolymer pastes [92]. In the case of fly ash and slag blends, the reaction at 27°C was dominated by the slag activation, whereas the reaction at 60°C was due to combined activation of fly ash and slag. The improvement in compressive strength of pastes with slag addition was attributed to its compactness of the microstructure [12]. The initiation of hardening in geopolymer concrete produced with sly ash and slag binder and activated with potassium silicate and potassium hydroxide was due to C-S-H / C-A-S-H formation and the hardening continued due to a rapid formation of a C-A-S-H, K-A-S-H and (Ca, K)-A-S-H depending on the availability of calcium ions and pH of the system. A slower dissolution rate of calcium ions effectively increased the compressive strength as rapid geopolymerization continues for a longer duration [93]. The low pH and limited calcium ion environment facilitated the polymerisation reaction between silicate and aluminate species in high calcium fly ash-based geopolymers producing NA-S-H gel [96]. Guo et al. [95] reported 63.4 MPa compressive strength of class C fly ash-based geopolymer paste showing the role of calcium participation in the strength development.

2.4.2.5 Concentration of sodium hydroxide (NaOH) solution

The concentration, expressed by molarity of the activating solution determines the resulting mechanical properties. While high NaOH additions accelerate chemical dissolution, it depresses ettringite and CH formation during reaction [97]. Reduction in the CH content resulted in superior strength and durability performance [98]. Furthermore, higher concentration (in terms of molarity) of sodium hydroxide solution results in a higher compressive strength of geopolymer concrete [37]. Additionally, the use of sodium hydroxide as an activator buffers the pH of pore fluids, regulates hydration activity and directly affects the formation of the main C-S-H product in geopolymer pastes. There is a linear relationship between NaOH concentration and the heat generation; however, there exists an inverse relationship between concentration and the time at which maximum hydration heat occurs [99].

2.4.2.6 Sodium silicate-to-sodium hydroxide liquid ratio

The addition of sodium silicate to the mix design improves mechanical properties beyond the ability of a hydroxide activator alone. However, care must be taken to regulate the ratio between each substance. Previous study indicated that the ratio of sodium silicate to sodium hydroxide plays a vital role on the development of mechanical properties of geopolymer concrete. The higher the mass ratio of sodium silicate-to-sodium hydroxide liquid, higher is the compressive strength of geopolymer concrete [73].

2.4.2.7 SiO_2 / Na_2O Ratio

The SiO_2 / Na_2O ratio is an important parameter in geopolymer design. It is well known that variations in the SiO_2 / Na_2O ratio significantly modifies the degree of polymerization of the dissolved species in the alkaline/silicate solution, therefore, determining the mechanics and overall properties of the synthesized gel product [63, 64]. Moreover, it is noted from the previous research that a high SiO_2 / Na_2O ratio (1.6 and 2.0) was used to synthesize a geopolymer, the compressive strength was higher than a certain maximum because more geopolymer precursors formed at the maximal strength [100]. Higher percentages of soluble silica in geopolymer systems retards dissolution of the ash material due to increased saturation of the ionic silica species and promotes the precipitation of larger molecular species, resulting in a stronger gel with an enhanced density [101].

2.4.2.8 Water-to-geopolymer solids ratio

The water content in the mixture played an important role on the properties of geopolymer binders [19]. The addition of any extra water in geopolymer mixtures improved the workability of the mixtures. However, the compressive strength of geopolymer concrete decreases as the ratio of water-to-geopolymer solids increases [102]. This trend is analogous to the well-known effect of water-to-cement ratio on the compressive strength of Portland cement concrete.

2.4.2.9 pH Level

The strength of the geopolymer concrete can be affected by the value of pH. The pH value with a range of 13–14 was found the most suitable condition for development of good mechanical strength [103]. The research also showed that an increase of the alkaline activator concentration directly raises the pH and consequently enhances the degree of reaction. Moreover, pH also plays a vital role for the viscosity of the geopolymer mixture. Lower pH value makes the mixture more stiff and viscous. On the other side higher pH makes the concrete more workable.

2.4.2.10 Raw Materials

Raw materials must constitute a large portion of Aluminum and Silica, inorganic non-metallic minerals and industrial waste, of which the main active ingredient is aluminum silicate. There are different kinds of raw materials that can be used to produce geopolymer cement, such as fly ash, red mud, metakaolin, natural pozzolan, blast steel slag, rice husk ash, and etc. In this study, the class F fly ash is used to form the geopolymer cement. Geopolymers possess different mechanical and thermal properties due to different raw materials, such as their variable chemical composition, particle size (fineness) and particle shape. Most of the recent studies are found focusing on the mechanical and thermal properties of the fly ash based geopolymers.

It is observed that a higher content of the glass phase will ensure a higher degree of geopolymerization, and thus resulting in a higher compressive strength. In addition, finer fly ash balls will lead to a relatively larger contacting surface area and hence higher reactivity can be guaranteed. Both the utilization of higher combustion temperatures and the grinding of the fly ash can make the fly ash balls much finer [65]. Further, a small portion of unburned coal in the fly ash will require a higher ratio of the alkali-activators to the fly ash, resulting in detrimental influence on the mechanical properties of the final geopolymer products.

2.5 STRUCTURAL PROPERTIES OF GEOPOLYMER CONCRETE

Joseph et al. (2012) studied influence of aggregate content on the behaviour of fly ash based geopolymer concrete and it was concluded that tensile strength of geopolymer concrete increased with increase in the total aggregate content. Further, as the total aggregate content varied from 60% to 75% (with constant fine aggregate to total aggregate ratio of 0.35), the split and flexural tensile strengths increased by 45.5% and 30.6%, respectively. Geopolymer concrete with total aggregate content of 70% by volume, ratio of fine aggregate to total aggregate of 0.35, NaOH molarity 10M, Na_2SiO_3 / NaOH ratio of 2.5 and alkali to fly ash ratio of 0.55 was prepared and cured for 24 hours at 100°C which gave an average cube compressive strength of 52 MPa after temperature curing (56 MPa after 28th day) [27].

Shaikh (2016) studied the effects of recycled coarse aggregates (RCA) on the mechanical and durability properties of fly ash geopolymer concrete. The mechanical properties were measured at 7 and 28 days. The inclusion of recycled coarse aggregate (RCA) as a partial replacement of natural coarse aggregates (NCA) in geopolymer concrete adversely affected its compressive and indirect tensile strengths and also the elastic modulus. Interestingly, the elastic modulus of geopolymer concrete containing RCA at 28 days was slightly lower than at 7 days. Further, maximum reduction in 7 and 28 days indirect tensile strength was about 23% and 16%, respectively at RCA content of 50%. Also, by increasing the RCA contents the compressive strengths decreased gradually, however, at very low rate e.g. only 15% and 16% at 7 and 28 days, respectively, for RCA content of 50%, which is slightly lower than conventional concrete produced with OPC and contained 50% RCA of same type [104]. This has further been confirmed by Xiao et al. (2005) [105].

Ryu et al. (2013) studied the mechanical properties of fly ash-based geopolymer concrete with alkaline activators and it was concluded that geopolymer concrete can be used instead of conventional concrete as it could achieve 44MPa compressive strength using heat curing [106].

Joe et al. (2014) studied the properties of the geopolymer concrete and the maximum compressive, flexural and split tensile strengths recorded were 40.5MPa, 5.45Mpa and 4.23MPa, respectively [107]. It was observed that strength increased with the increase in NaOH concentration.

Aravindan et al. (2015) studied the behaviour of geopolymer concrete by replacing fly ash with slag using 12M hydroxide solution. A remarkable compressive strength of 85MPa was observed. Similarly, split and flexural strengths were 2.7MPa and 6MPa, respectively, [108].

Therefore, it is clear from the above discussion that efforts have been made in last three decades on the development and manufacturing of fly ash based geopolymer concrete. Efforts have been made in some studies to develop the geopolymer concrete cured at ambient temperature by partially replacing the fly ash with GGBS, cement or other materials which were rich in calcium oxide. However, the structural properties of RHA and GGBS based geopolymer concrete have not been studied.

2.6 DURABILITY OF GEOPOLYMER CONCRETE

One of the major problems associated with OPC concrete is its long-term durability which had always been an issue against aggressive environments. The deterioration of concrete is usually assessed for atmospheric carbonation, sulphate and chloride induced corrosion, alkali-silica reaction and freeze–thaw attack. A few studies are available on durability of geopolymer concrete involving these aspects and have been summarized as under.

2.6.1. Alkali-silica reaction

Alkali-silica reaction (ASR) causes gradual but severe deterioration of hardened Portland cement concrete in terms of its strength loss, cracking, volume expansion etc. It involves the reaction between the hydroxyl ion present in the pore solution and reactive silica of the aggregate. In general terms, the reactions will proceed in stages, with the first stage being the hydrolysis of reactive silica by hydroxyl ions to form alkali-silica gel and a later secondary overlapping stage being the absorption of water by the gel, which results in expansion and disintegration of concrete [109].

In geopolymer concrete, the un-utilised alkali after geopolymerization of alumino-silicates is expected to react with the silica of the aggregates causing disruption of their siloxane bridges. It is reported that geopolymer mortars using aggregates of different reactivities expanded less than the corresponding Portland cement mortars [110]. The geopolymer mortars appeared to be sound without any surface cracking. The cause of expansion in slag-based geopolymer mortars was the formation of sodium calcium silicate hydrate reaction product with rosette-type morphology [111]. Contrary to this, there was no significant expansion in fly ash based geopolymer mortars. However, the expansion was less than 0.1% limit prescribed in ASTMC1260-07 after 16 days (Fig. 2.2) when the combination of fly ash and slag was used.

At 90 days exposure, these mortars failed to meet the specified criteria. Increasing slag content in fly ash/slag mix increased the expansion of resulting systems [87]. ASR has also been claimed to be helpful in providing a strong bond at the paste-aggregate interface, thus resulting in higher tensile strength of GPC [66].

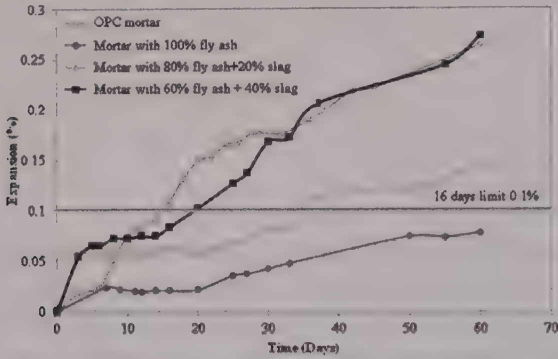


Fig. 2.2: Alkali-silica reaction in various geopolymer and OPC mortars under an accelerated condition (1 M NaOH) at 80°C [87].

Patil et al. [110] indicated that sandstone, quartz and limestone aggregates in geopolymer concrete were not prone to ASR. During accelerated mortar bar test, a slight expansion was noticed because of re-initiation of the geopolymerization process of unreacted fly ash particles leading to lower porosity and higher strength. The lower sensitivity of reactive aggregates in GPC provides economic advantages in areas where high quality deposits of aggregates have been depleted.

2.6.2. Effect of acid attack

The acid resistance of geopolymer pastes/concrete was studied by several authors [77, 112-116]. The extent of degradation dependent on the concentration of acid solution and period of exposure. Davidovits et al. [66] indicated that metakaolin-based geopolymer pastes showed only 7% mass loss when sample was immersed in 5% H_2SO_4 for 30 days. It was also reported that fly ash-based geopolymer pastes retained a dense microstructure even after 3 months exposure in HNO_3. Temuujin et al. [112] concluded that acid and alkaline resistances of fly ash-based geopolymer strongly depended on its mineralogical composition. High solubility of Al, Si and Fe ions was obtained in both strong alkali and acid solutions. The performance of fly ash-based geopolymer pastes when exposed to 5% acetic acid and 5% H_2SO_4 solutions was superior to ordinary Portland cement pastes. The deterioration in pastes was connected to depolymerisation of the alumino-silicate network and formation of zeolites [77].

Wallah and Rangan [71] found that the reduction in compressive strength of fly ash-based GPC in 0.5% H_2SO_4 solution was 20% after 12 months exposure. The reduction further decreased with the concentration of H_2SO_4 as it was ~52% and ~65%, respectively, when samples exposed to 1% and 2% H_2SO_4 solution. Pitting and erosion on the surface of the concrete were also observed. The loss in strength of concrete is mainly due to the degradation in the geopolymer matrix rather than the aggregate. They concluded that the acid resistance of GPC was superior to conventional concrete. Ariffin et al. [113] exposed GPC made with a blend of pulverized fuel ash and palm oil fuel ash in 2% solution of sulphuric acid for 18 months. The weight loss in GPC was 8% while conventional concrete exhibited 20% weight loss. The strength reduction in GPC was 35% in 18 months as against 68% strength loss in conventional concrete after 30 days and was deteriorated severely after 18 months. The C-S-H could have severe deleterious effects on conventional concrete while N-A-S-H gel appeared to have little effect on the structure of GPC.

Bakharev et al. [114] found that slag-based GPC (40 MPa) exhibited ~33% reduction in strength compared to 47% in OPC concrete when exposed in acetic acid solution (pH 4) for 12 months. The slag particles and low calcium C-S-H with average Ca-Si ratio of 1 were more stable in the acid solution than in the constituents of the OPC pastes., The strength loss had been approximately ~11% compared to 36.2% for conventional concrete when immersed in 2% H_2SO_4 solution.

2.6.3. Effect of sulphate attack

Several attempts [23, 117] have been made to study sulphate resistance of GPC. The deterioration in concrete was evaluated in terms of its visual appearance, weight loss and change in compressive strength. Fly ash-based geopolymer pastes did not deteriorate significantly, under the influence of water, sodium sulphate (4.4%) and in sea water [115]. Only some fluctuations in flexural strength was observed between 7 days and 3 months exposures. The least strength change was observed in the pastes exposed in the 5% Na_2SO_4 and 5% $MgSO_4$ solutions while most significant deterioration was observed in the 5% mixed sulphate solution (Na_2SO_4 + $MgSO_4$) after 5 months exposure [78]. In fly ash and slag based system, the extensive physical deterioration of matrix was observed during immersion in $MgSO_4$ solution after 3 months exposure but not in Na_2SO_4 solution. The calcium sulphate dehydrate formed in matrix was identified as being particularly damaging to the materials in $MgSO_4$ [116]. In the case of Na_2SO_4 solution, only exposition of grains was clearly visible while in $MgSO_4$ solution, both exposition of grains and dissolved alumino-silicate matrix were observed showing severity of $MgSO_4$ attack [87]. The deterioration was considered mainly due to the destruction of alumino-silicate skeleton, liberation of silicic acid, leaching of sodium ion etc. These reactions seem to have significant effect on the mechanical strength. The fly ash based geopolymer concrete prepared with NaOH activator had the best performance over those made with a synergistically used sodium silicate and NaOH-KOH activators, which is attributed to its stable cross-linked alumino-silicate polymer structure. Hardjito et al. [37] observed that there were no significant effects of 5% Na_2SO_4 solution in the compressive strength, the weight loss and the dimension of fly ash-based GPC after 3 months exposure.

Rajamane et al. [117] reported sulphate resistance of fly ash-based GPC for 3 months in 5% Na_2SO_4 and 5% $MgSO_4$ solutions. The weight loss in samples was 2.4% only. There was 2–29% loss of compressive strength as compared to 9–38% in the conventional concrete. The deterioration of conventional concrete can be attributed to the formation of expansive gypsum and ettringite which is the cause expansion, cracking and spalling in the concrete. Contrary to this, GPC in general does not contain $Ca(OH)_2$ and mono sulpho-aluminate in the matrix to cause expansion.

2.6.4. Carbonation and permeability

Bernal et al. [118] studied slag-metakaolin based GPC (w-b ratio 0.47) under an accelerated carbonation test using CO_2 concentration of $3.0 \pm 0.2\%$ at 20°C for 28 days. They found that the compressive strength decreased monotonically as the carbonation proceeded. The relationship between the pore volume and extent of carbonation of slag-metakaolin based GPC was similar with samples with different percentages of metakaolin contrary to the slag-based samples. This suggested that porosity was not the only parameter controlling the strength loss of the carbonated binder. There must be a convoluting effect due to the binder gel chemistry, which determines the residual level of strength after an accelerated carbonation.

Olivia and Nikraz [96] reported lower water permeability ($2.46–4.67 \times 10^{-11}$ m/s) of GPC (activator-fly ash ratio, 0.30–0.40 cured at 60°C for 24 h) than the conventional concrete due to its denser paste and smaller pore inter-connectivity. They also reported that the water-geopolymer solids ratio was the most influential parameter that affected the properties of GPC.

Bondar et al. [119] studied the oxygen and chloride permeability of alkali-activated concrete made with the Iranian natural pozzolan (Taftan andesite and Shahindej dacite). They concluded that alkali-activated natural pozzalona concrete had 10–35% lower oxygen permeability at normal curing conditions for 90 days compared with the conventional concrete. The rapid chloride permeability test gave high values for the alkali-activated concrete. This was probably due to the very high alkali ion concentration in the pore solution promoting higher electrical conductivity in the GPC. This effect seems to reduce with age due to a change in the porosity of the GPC microstructure.

2.6.5. Corrosion of steel reinforcement

Half-cell potential is a technique used to detect the state of reinforcement without disturbing the structures. This is important because the intensity of corrosion of steel in concrete is generally known only after the concrete has cracked or disrupted. Various studies [96, 120] were reported to estimate the corrosion potential of steel within the GPC as per ASTM C876. Olivia and Nikraz [96] reported that the half-cell potential of GPC was lower than the specified value of -404 mV mentioned in the Standard for severe corrosion after 91 days.

Sathia et al. [121] also reported half-cell potential up to -300 mV which showed a probable corrosion indication due to the lower pH of concrete during the half-cell potential measurement. Accelerated corrosion results showed that GPC mixes exhibited low level corrosion activity and time to failure that was 3.86–5.70 times longer than those of the conventional concrete. Under impressed voltage, a crack appeared suddenly in the concrete when time to failure was reached and this was followed immediately by high current reading. The large amounts of fly ash and alkaline activators in the GPC mix increased the availability of OH ions that can produce high electrical resistance at high impressed voltage. This enhanced the cathodic reaction and reduced the rate of corrosion, which in turn, reduced the tensile stress of the specimens, thus decreasing the risk of cracking and clearly extending the time to failure [96].

Reddy et al. [120] compared the durability of GPC with that of conventional concrete exposed to marine environment for a period of 21 days. The initial corrosion current measured for GPC (71–91 mA) was much lower than that of conventional concrete (772 mA). The conventional concrete specimens initially recorded decrease in the current but later started increasing while the GPC current never showed significant increase.

2.7 MIX DESIGN OF GEOPOLYMER CONCRETE

Hardjito et al. (2005) in their study focussed to identify the salient parameters those influenced the mixture proportions using low calcium fly ash-based geopolymer concrete [32]. They followed the current practice used in the mix proportioning of conventional concrete. Based on the experimental work reported in their study, the following conclusions were drawn:

1. Higher concentration (in terms of molar) of sodium hydroxide solution resulted in higher compressive strength of fly ash-based geopolymer concrete.
2. Higher the ratio of sodium silicate-to-sodium hydroxide ratio by mass, higher was the compressive strength of fly ash-based geopolymer concrete.
3. As the curing temperature in the range of 30°C to 90°C increased, the compressive strength of fly ash-based geopolymer concrete also increased.
4. Longer curing time, in the range of 4 to 96 hours (4 days), produced higher compressive strength of fly ash-based geopolymer concrete. However, the increase in strength beyond 24 hours was not significant.
5. The addition of naphthalene sulphonate-based super plasticiser, up to approximately 4% of fly ash by mass, improved the workability of the fresh fly ash-based geopolymer concrete; however, there was a slight degradation in the compressive strength of hardened concrete when the super plasticiser dosage greater than 2%.
6. The slump value of the fresh fly-ash-based geopolymer concrete increased with the increase of extra water added to the mixture.
7. The Rest Period, defined as the time taken between casting of specimens and the commencement of curing, of up to 5 days increases the compressive strength of hardened fly ash-based geopolymer concrete. The increase in strength was substantial in the first 3 days of rest period.
8. The fresh fly ash-based geopolymer concrete was easily handled up to 120 minutes without any sign of setting and without any degradation in the compressive strength.
9. As the H_2O-to-Na_2O molar ratio increased, the compressive strength of fly ash-based geopolymer concrete decreased.
10. As the ratio of water-to-geopolymer solids by mass increased, the compressive strength of fly ash-based geopolymer concrete decreased.

11. The effect of the Na_2O-to-Si_2O molar ratio on the compressive strength of fly ash-based geopolymer concrete was not significant.

12. The compressive strength of heat-cured fly ash-based geopolymer concrete did not depend on age.

13. Prolonged mixing time of up to sixteen minutes increased the compressive strength of fly ash-based geopolymer concrete.

14. The average density of fly ash-based geopolymer concrete was similar to that of conventional concrete.

Aleem et al. (2012) reported the optimum mix design for geopolymer concrete in terms of fine aggregate to coarse aggregate ratio and by keeping the fly ash content same [33]. Concrete cubes of size 150 mm were prepared and cured under steam curing for 24 hours. The compressive strength was recorded at 7 days and 28 days. In their study, four-trial mixes (1:1.3:3.10, 1:1.4:3.20, 1:1.5:3.30, 1:1.6:3.40) were taken based upon the earlier studies done on conventional concrete and optimum mix was selected after results. The maximum compressive strength achieved in their study was 53.33 MPa using 1:1.5:3.30 ratio and after 28 days. The increase in percentage of fine aggregates and coarse aggregates increased the compressive strength up to the optimum level. This might be due to the high bonding between the aggregates and alkaline solution.

Ramujee et al. (2013) in their study attempted to develop the mix design for geopolymer concrete in low (G20), medium (G40) and higher (G60) grades [34]. Further, relative comparison was done with equivalent mix proportions of grades of conventional concretes in both heat cured (60°C) and ambient cured (27°C) conditions. Seven different mixes by keeping the S.S-S.H and fine aggregate-total aggregate ratios equals to 2.5 and 0.35, respectively, for each grade (i.e. low, medium and higher) were cast, tested and optimized. It was found that the results obtained were in agreement with mix design reported. The design parameters like alkaline liquid to fly ash, water to geopolymer solids ratios and different NaOH molarity were proposed to develop the Geopolymer concrete in lower, medium and high grades concrete. An attempt has been made in this research to design the mix for geopolymer concrete.

Madheswaran et al. (2013) in their research tried to achieve the strength for various grades of geopolymer concrete with varying molarity [122]. In order to meet the performance criteria in-terms of required workability and compressive strength, factors influencing the geopolymer concrete were selected based on the previous studies. Different molarities of sodium hydroxide solution (3M, 5M and 7M) were taken to prepare different mixtures. GPC mix formulations with compressive strength ranging from 15 to 52MPa was developed with the help of slag and fly ash as a binder in the matrix. Major conclusion of this study was that mixture components of GPC can be designed using the tools available for the conventional concrete.

Patankar et al. (2015) in their research attempted to select the suitable ingredients of geopolymer concrete to achieve desire strength at required workability. A mix design procedure was proposed on the basis of quantity and fineness of fly ash, quantity of water, grading of fine aggregate, fine to total aggregate ratio [123]. Water-to-geopolymer binder ratio of 0.35, alkaline solution-to-fly ash ratio of 0.35, sodium silicate-to-sodium hydroxide ratio of 1.0 by mass and 13M NaOH were fixed on the basis of workability and cube compressive strength. It was observed that the results of workability and compressive strength were well match with the required degree of workability and compressive strength. Further, geopolymer concrete of grade M20, M25, M30, M35 and M40 were taken for reference.

Junaid et al. (2015) developed a systematic approach for selecting mix proportions for alkali activated fly ash-based geopolymer concrete (GPC). The research was carried out with detailed examples of mix designs for various strengths. A formal framework to perform a mix design for alkali activated Class F fly ash geopolymer concretes was developed along with useful design aids for a wide range of strength and workability requirements for practical applications. In this approach, the parameters; W-GPS, AL-FA, AL-W were identified as the most significant ratios whose relations determine the quantities of the mix and the performance of the hardened product with confidence. A step by step procedure illustrated in their research with the aid of examples has served to show the application of the method and its ability to produce consistent results when heat curing is adopted.

Pavithra et al. (2016) in their research made an effort to develop a mix design methodology for GPC with the main focus on achieving better compressive strength in an economical way for different alkaline solutions to binder proportions [36]. Mix design was proposed for various AAS-FA ratios ranging from 0.4 to 0.8, and the 28 days compressive strength as high as 54 MPa had been noticed. The findings of the study suggested that, using the proposed method GPC can be produced for a specific strength by employing the corresponding AAS-FA ratio obtained from the modified ACI strength vs. w/c ratio curve. GPC can also be produced for a specific AAS-FA ratio to achieve the corresponding strength. It had been found that, GPC follow similar trend to that of normal concrete in the strength aspect where the strength decreases with the increase in the fluid content.

2.8 STRESS-STRAIN BEHAVIOUR

Hardjito et al. (2005) in their study conducted tests on the geopolymer concrete specimens. The tests on 100 x 200 mm concrete cylinders were performed by using the displacement-control mode available in the test machine. The stress-strain relations of fly ash-based geopolymer concrete in compression fitted well with the expression developed for conventional concrete, with the strain at peak stress in the range of 0.0024 to 0.0026. These values were similar to those reported for conventional concrete. Further, it was predicted that stress-strain relationship for fly ash based geopolymer concrete can be predicted by using the equations developed for conventional concrete.

Haider et al. (2013) in their study established that the stress-strain behaviour of the geopolymer concrete was not much different from conventional concrete. In this study, it was observed that geopolymer concrete underwent less deformation under compressive loading in initial stage rather than at failure. In their study, stress-strain characteristics of geopolymer concrete under various levels of confining stresses (0 to 35MPa) had been studied using the triaxial laboratory tests. Based on the results they proposed a constitutive model describing the behaviour of geopolymer concrete in terms of stress-strain which shows many similarities to conventional concrete [124].

Ganesan et al. (2014) in their study focussed to study the stress-strain behaviour of confined (using 6mm HYSD bars) and unconfined geopolymer concrete. Stress-strain curves of GPC and conventional concrete specimens with various percentage (1.36%, 2.05% and 4.1%) of spiral confinement were tested and it was found from their study that stress-strain behaviour was almost similar for both the GPC and conventional concrete. Further, it was found that at initial stage, the deformation of the unconfined GPC specimens increased at slower rate than that of conventional concrete but at later stage (after 80% of peak stress) deformation was faster than PCC specimens [125]. It was concluded that GPC deformation rate was higher because of the presence of micro cracks at peak stresses. Also, a stress-strain model proposed by Mander et al. (1988) for PCC with confinement could be used for confined GPC with a modification in the curve fitting factor. Further, it was concluded from their study that GPC and conventional concrete had similar brittle behaviour [125, 126].

Manjunatha et al. (2014) represented the typical stress-strain and the normalized stress-strain relationships, respectively, for geopolymer concrete. The typical and the normalized stress-strain relationships were found to be non-linear. Therefore, the secant value of the modulus of elasticity was determined from the normalized stress-strain curve at stress level of one-third of the average ultimate strength as per the provision of IS 516-1959. The secant modulus of elasticity and the peak strain of the geopolymer concrete considered in the study had been calculated as 16.68 GPa and 0.0028, respectively. Arriving at standard stress-strain relationships for geopolymer concrete and their incorporation in the design equations were going to be challenging tasks in view of large number of additional influencing factors such as water-binder, Na_2SiO_3-NaOH and fly ash-slag ratios etc. [127].

Junaid et al. (2015) in their study developed the stress-strain model of the gepolymer specimens prepared at heat temperature (ranging from 100° to 800°C) and compared it with the other models on geopolymer concrete. Several existing analytical stress-strain models were studied and compared with the GPC experimental data; among them those proposed by Collins et al. [128] and Sargin and Handa [129]. It was found that all the GPC specimens experienced an initial loss in strength and stiffness properties when exposed to high temperatures still the values were on higher side when compared to conventional concrete. It was concluded in this study that geopolymer concrete behaviour was similar to that of conventional concrete but generalised analytical model for the stress-strain relationship of GPC at higher temperatures is not yet possible.

Summary: - Research is going on from last 30 years on the geopolymer concrete and the literature available relevant to the topic is presented in this chapter. The effect of mix design and other parameters on the mechanical properties of the GPC are gathered and critically discussed. Though there is sufficient evidence that geopolymer concrete have several advantages over conventional concrete but it has been identified that there is a gap in the literature in the area of geopolymer concrete for waste materials like FA, RHA and GGBS and which are suitable for ambient curing. In addition to that mix design procedure is not available for the same materials at ambient and heat curing which satisfy the workability and strength requirements. Therefore, experimental work has been designed to study the design and production of geopolymer concrete covering different temperatures, molarities of NaOH and by using different waste materials.

CHAPTER - 3

MATERIALS, PREPARATIONS AND METHODS

3.1 INTRODUCTION

Based upon the available literature and the identified gap, ingredients procured and preparation was done to develop the geopolymer concrete using different variables. In this Chapter, details of different materials used, method of casting, curing of specimens and details of methodology adopted are presented.

3.2 MATERIALS

3.2.1 Fly Ash

Two types of fly ash were used in this study. Geopolymer concrete was developed by using processed and unprocessed types of fly ash during the preliminary laboratory study. Low calcium (calcareous- Processed) fly ash with specific gravity 1.95 confirming to IS : 3812 - 2013 [130] was used in current study which was provided by Ultra Tech RMC Plant, Panchkula, Haryana. Un-processed fly ash was procured from Panipat Thermal Power Plant. Fly ash was used as a source of alumina-silicate. The image of the processed fly ash particles along with SEM micrograph is shown in Fig. 3.1. The physical and chemical properties of fly ash is shown in Table 3.1. Further, it was analysed using X-ray fluorescence spectroscopy (XRF) which showed the presence of quartz, mullite and calcite type compounds as shown in Fig. 3.2.

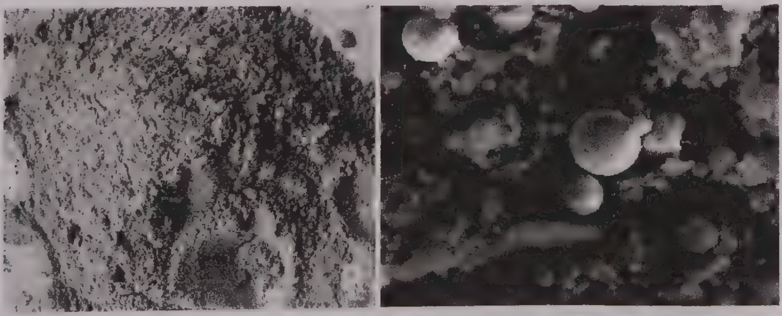


Fig. 3.1: Photograph and SEM image of fly ash

Table 3. 1: Chemical composition and physical properties of fly ash

Composition (%)	**Processed Fly Ash**	**Un-Processed Fly Ash***	**IS 3812-2013 requirement [130]**
Silica + alumina + iron oxide ($SiO_2 + Al_2O_3 + Fe_2O_3$): wt%	95.91	91.25	70.0 (Min)
Silica (SiO_2) : wt%	62.55	56.90	35.0 (Min)
Calcium Oxide (CaO) : wt%	0.87	0.85	Not specified
Magnesia (MgO) : wt%	0.39	1.21	5.0 (Max)
Sulphur trioxide (SO_3) : wt%	1.32	1.38	3.0 (Max)
Sodium oxide (Na_2O) : wt%	0.46	0.52	1.5 (Max)
Total chlorides : wt%	0.05	0.025	0.05 (Max)
Loss on ignition : wt%	0.52	1.85	5.0 (Max)
Fineness-specific surface, m^2/kg	321.7	255.2	320 (Min)

X-ray Diffraction (XRD) analysis and particle size distribution (PSD) curve of fly ash are depicted in Fig.s 3.2 and 3.3, respectively.

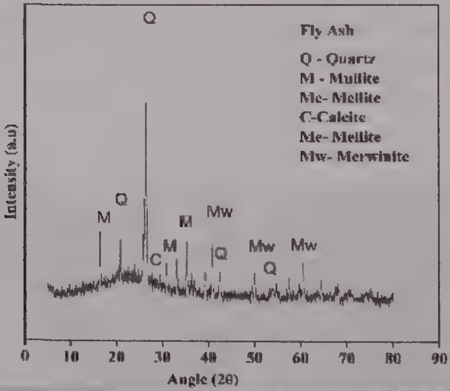


Fig. 3.2: XRD spectrum of fly ash

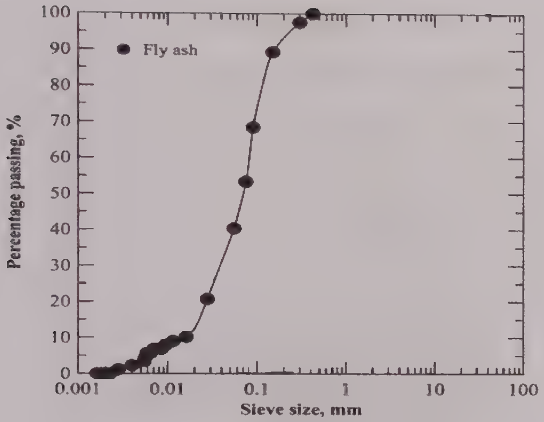


Fig. 3.3: Grading properties of fly ash

3.2.2 Rice Husk Ash (RHA)

Rice husk ash is carbon neutral and a kind of super pozzolon green product which can be used to make special concrete mixes. There is a growing demand for fine amorphous silica in the production of special cement and concrete mixes, high performance concrete, high strength and low permeable concrete for use in different projects. The RHA shown in Fig. 3.4 used in this study was purchased from a commercial supplier N K Enterprises, Orissa, India and the compositions of RHA are given in Table 3.2. The X-ray diffraction analysis is depicted in Fig. 3.5.

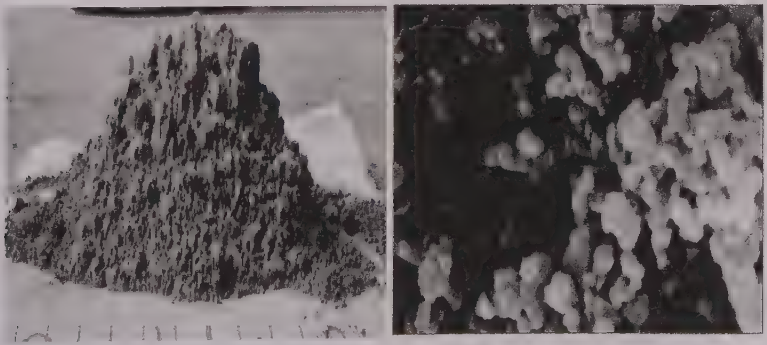


Fig. 3.4: Photograph and SEM image of rice husk ash

Table 3. 2: Chemical composition of rice husk ash

Composition (%)	**Rice Husk Ash**
Silica (SiO_2) : wt%	92.96
Alumina (Al_2O_3) : wt%	0.14
Iron oxide (Fe_2O_3) : wt%	0.05
Titanium dioxide (TiO_2) : wt%	0.01
Calcium Oxide (CaO) : wt%	0.45
Magnesia (MgO) : wt%	0.19
Sulphur trioxide (SO_3) : wt%	1.32
Sodium oxide (Na_2O) : wt%	0.29
Potassium oxide K_2O : wt%	2.38
P_2O_5 : wt%	0.29
Loss on ignition : wt%	3.24
Specific gravity	2.13
Specific surface area m^2/kg	355

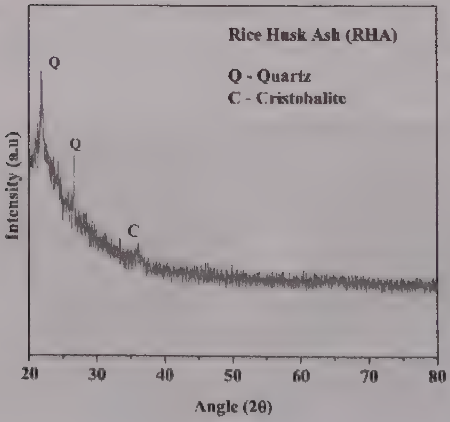


Fig. 3.5: XRD spectrum of rice husk ash

3.2.3 Ground Granulated Blast Furnace Slag (GGBS)

GGBS is a glassy, granular material which is a by-product from a blast furnace. The chemical composition of a slag varies considerably depending on the composition of the raw materials in the iron production process. It is a non-metallic product, consisting of silicates and alumino-silicates of calcium and other bases, developed in a molten condition simultaneously with iron in a blast furnace. The GGBS shown in Fig. 3.6 used in this study was purchased from a commercial supplier (JSW cements) and the compositions of GGBS are given in Table 3.3. The X-ray diffraction analysis is depicted in Fig. 3.7.

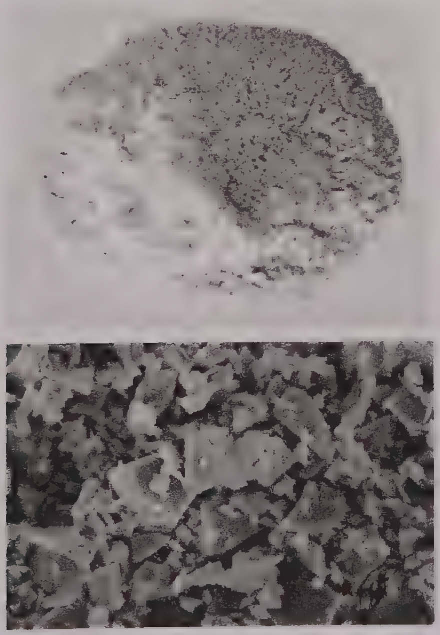


Fig. 3.6: Photograph and SEM image of GGBS

Table 3. 3: Chemical composition of GGBS

Composition (%)	GGBS
Iron oxide (Fe_2O_3): wt%	2.0
Silica (SiO_2) : wt%	37.50
Alumina (Al_2O_3)	13.80
Calcium Oxide (CaO) : wt%	42.2
Magnesia (MgO) : wt%	3.70
Sulphur trioxide (SO_3) : wt%	0.20
Loss on ignition : wt%	0.60
Specific gravity	2.60
Specific surface area m^2/kg	375

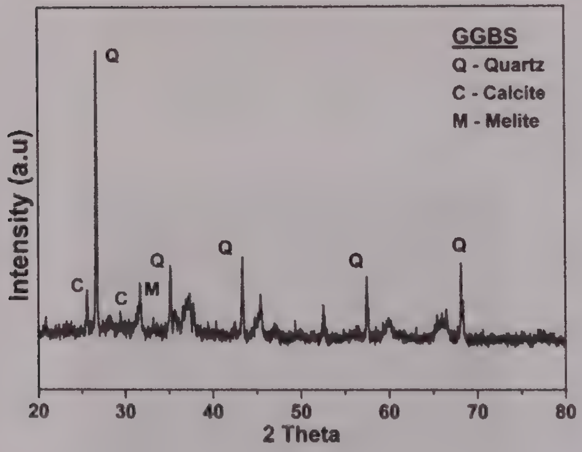


Fig. 3.7: XRD spectrum of GGBS

3.2.4 Fine Aggregates

Locally available river sand was used. The sand was obtained from Yamuna River and was mixed with the coarse sand in 50 : 50 proportions to achieve the grading of sand, which conformed to Zone II as per IS 383 : 1970 [131]. The sand used was cleaned from all inorganic impurities and the sand, which passed through 2.36 mm sieve and retained on 150 micron had been used. The sieve analysis of sand is presented in Table 3.4 and physical properties of fine aggregate in Table 3.5. Fineness modulus of fine aggregates was 2.83 with a specific gravity of 2.60 and water absorption 1.5%. The grading curve of the fine aggregates is depicted in Fig. 3.8.

Table 3. 4: Sieve analysis of fine aggregate

IS sieve size	Wt. retained (gms)	Cumulative wt. retained (gms)	Cumulative % retained	Cumulative % passing
4.75 mm	-	-	-	100
2.36 mm	65	65	6.5	93.5
1.18mm	210	275	27.5	72.5
600 µm	318	593	59.3	40.7
300 µm	300	893	89.3	10.7
150 µm	107	1000	100	-
pan	-	-	-	-
Total	1000		282.6	
Fineness Modulus = 282.6/100 = 2.826				

Table 3. 5: Physical properties of fine aggregate

S.No	Characteristics	Tested values
1.	Specific Gravity	2.60
2.	Fineness Modulus	2.82
3.	Water Absorption (%)	1.50
4.	Moisture Content (%)	0.50
5.	Grading	Zone II

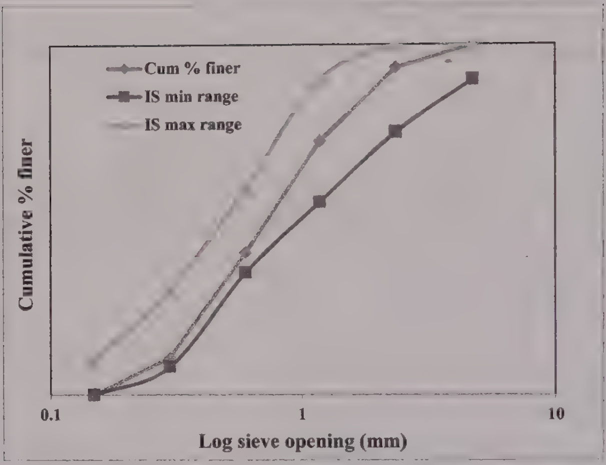


Fig. 3.8: Grading properties of fine aggregate

3.2.5 Coarse Aggregates

Coarse aggregates used in this study comprised of 14 mm, 10 mm and 7 mm downgraded in the saturated surface-dry (SSD) condition. Physical properties of coarse and fine aggregates confirmed to IS 383-1970 [132] , while fine aggregate used are crushed sand and graded as conforming to IS: 2386 (Part I)-1963[133]. The sieve analysis and physical properties of coarse aggregates are presented in Tables 3.6 and 3.7, respectively. The grading curve which confirmed the requirement of Indian standard is shown in Fig. 3.9.

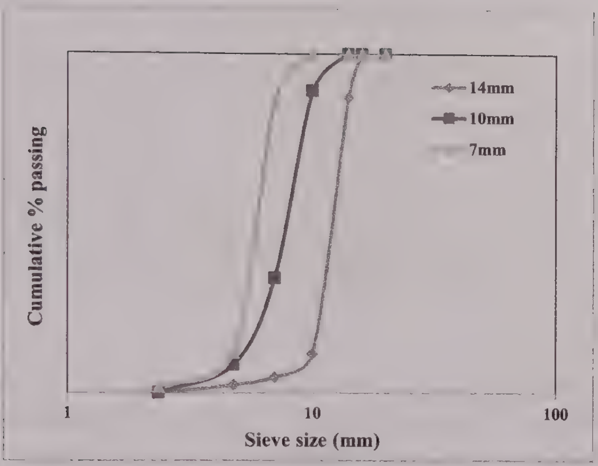


Fig. 3.9: Grading properties of coarse aggregate

Table 3. 6: Sieve analysis of coarse aggregate

IS sieve size (mm)	Cumulative % passing (14mm)	Cumulative % passing (10mm)	Cumulative % passing (7mm)	Combined* cumulative % passing
20	100	100	100	100
16	100	100	100	100
14	87.33	100	100	94.29
10	11.67	89.28	100	56.49
7	4.77	33.93	86.53	31.28
4.75	2.37	8.36	11.86	6.36
2.36	0.00	0.00	1.73	0.34
*14mm@45% + 10mm@35% + 7mm@20%				

Table 3. 7: Physical properties of coarse aggregate

S.No	Characteristics	Tested values
1.	Specific Gravity	2.66
2.	Fineness Modulus	7.10
3.	Water Absorption (%)	0.80
4.	Moisture Content (%)	Nil
5.	Texture	Rough

3.2.6 Water

The mixing water should be clean, fresh and potable. The water should be relatively free from organic matter. Water as available from the tap which confirmed the requirements of IS 456 for construction purpose was used for mixing the ingredients of concrete and curing the specimens [134].

3.2.7 Alccofine 1203

Alccofine 1203 (AF) as shown in Fig. 3.10, a low calcium silicate micro fine material, which is a refined form of ground granulated blast furnace slag (GGBS) was obtained from Ambuja Cements Ltd, Andheri East, Mumbai. Alccofine is used in high-performance concrete as it improves the concrete properties in both fresh and hardened states [135, 136]. Chemical compositions and physical properties of Alccofine 1203 used are given in Table 3.8. The X-ray diffraction analysis is depicted in Fig. 3.11.

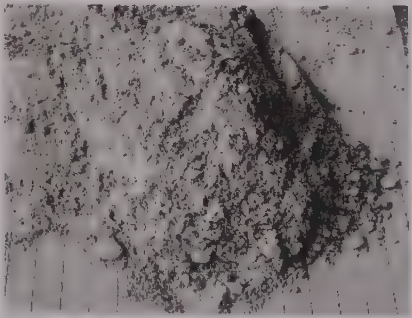


Fig. 3.10: Photograph of alccofine 1203

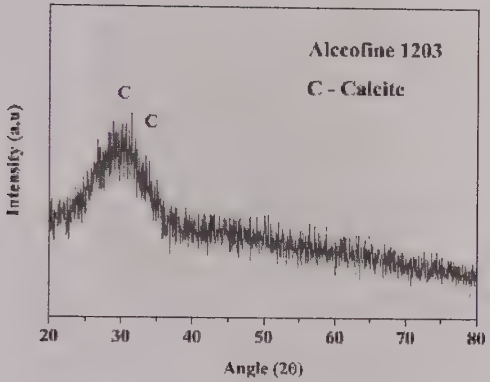


Fig. 3.11: XRD spectrum of alccofine

Table 3. 8: Chemical composition and physical properties of Alccofine 1203

Chemical Composition		Physical Properties		
Constituents	**Composition (%)**	**Physical Property**		**Results**
Fe_2O_3	1.20	Bulk Density (kg/m^3)		680
SO_3	0.13	Specific Gravity		2.70
SiO_2	35.30	Particle Size Distribution (in micro metre)	d10	1.8
MgO	8.20		d50	4.4
Al_2O_3	21.40		d90	8.9
CaO	32.20	Specific Surface Area		12000 cm^2/gm

3.2.8 Sodium Silicate

In this study, Sodium silicate in the form of heavy syrup was used as an alkaline activator which plays important role in the geopolymerisation process. Sodium silicate solution (Na_2SiO_3) with SiO_2-Na_2O between 1.90 and 2.01 were procured commercially. The weight analysis of this material as given by the supplier was:- Na_2O : 14.7%, SiO_2 : 29.4%, Water : 55.9%. The specifications of the sodium silicate shown in Fig. 3.12, are as shown in Table 3.9.

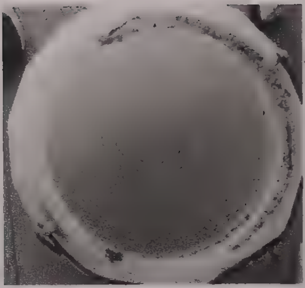


Fig. 3.12: Photograph of sodium silicate solution

Table 3.9: Sodium Silicate specifications

Item	**Specification**
Color	Color-less / cloudy
Density, gm/cm^3	1.45-1.55
Total solids content, by mass %	45:52

3.2.9 Sodium Hydroxide

Sodium hydroxide (NaOH) in the form of pellets with 98% purity was procured commercially. The solution was prepared by dissolving the pellets into the water at specified concentration in terms of molars, M, for the concrete and shown in Fig. 3.13.

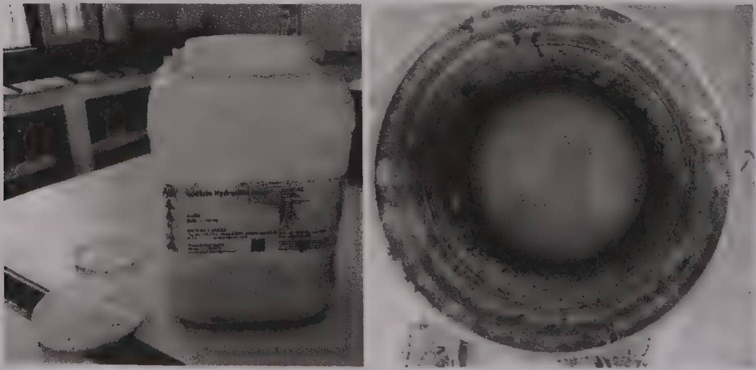


Fig. 3.13: Photograph of sodium hydroxide pallets and solution

3.2.10 Superplasticizer

Sodium silicate (SS) and sodium hydroxide (SH) solutions are more viscous than water, as such their use makes the GPC more cohesive and sticky than conventional concrete [137]. Therefore, in order to improve the workability of the fresh geopolymer concrete, a Naphthalene Sulphonate based water reducing superplasticizer confirming to IS 9103:1999 [138] was used.

3.3 PREPARATION OF TEST SPECIMENS

3.3.1 Preparation of Sodium Hydroxide

In all the prepared mixes, sodium hydroxide (NaOH) was used as an alkaline activator. The sodium hydroxide solution was prepared by dissolving the flakes or the pellets in the distilled water. The mass of NaOH solids in a solution varied depending on the concentration of the solution expressed in terms of molar, M. For instance, NaOH solution with a concentration of 8M consisted of 8 x 40 = 320 grams of NaOH solids (in flake or pellet form) per litre of the solution, where 40 was the molecular weight of NaOH. The mass of NaOH solids was measured as 262 grams, 361 grams, 444 grams per kg of NaOH solution of 8M, 12M and 16M concentration, respectively. The NaOH was prepared 24 hours prior to casting of the samples. The details of solution are as given in Table 3.10

Table 3.10: Mass of NaOH pellets and water for making 1kg of NaOH solution [31]

Molarity	Mass of NaOH (gms)	Mass of water (gms)
8M	262	738
12M	361	639
16M	444	556

3.3.2 Preparation of Alkali Solutions

Sodium silicate solution and sodium hydroxide solution were chosen as the alkaline liquid. Sodium-based solutions were chosen because they were cheaper than Potassium-based solutions. The NaOH solution was first prepared at the required molarity as explained in 3.3.1. The details about the method of making NaOH with the required molarity has been presented in 3.3.1. The alkali solutions as detailed above for geopolymer concrete were mixed and the reaction in between was exothermic. Different researchers have proposed different methods of mixing of alkali solutions. Further, some investigators premix the alkali solutions 24 hours before mixing with the other constituents for making GPC [13, 102, 139], others [119, 140] recommend adding the alkali solutions separately during the dry mixing itself. For the present study, the alkali solution was first prepared by thoroughly mixing the NaOH and Na_2SiO_3 solutions 2 hours prior to its use so that all the heat was evolved. The prepared solution was as shown in Fig. 3.14.

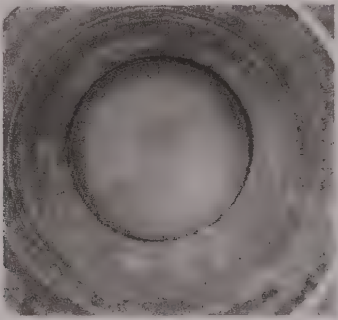


Fig. 3.14: Photograph of prepared alkaline solution

3.3.3 Preparation of Superplasticizer and Water

Superplasticiser and extra water was mixed properly for about 5 minutes and 30 minutes, respectively, prior to final mixing of the all ingredients of the GPC into the pan mixture.

3.3.4 Inclusion of Alccofine

Based on the preliminary laboratory results on the fly ash based geopolymer concrete it was concluded that required workability and strength at ambient curing could not be achieved with fly ash itself. Therefore, applicability of geopolymer concrete was restricted. As had been pointed out in 3.2.6, alccofine improves the properties of concrete while in fresh and hardened state, so, it was as such decided to include the alccofine into geopolymer concrete. Initially, different dosages of alccofine were tried starting from 0% to 20% and it was noticed from the results that strength and workability improved. Therefore, to produce the effective and economical concrete, it was decided to keep the amount of alccofine as 10% of the fly ash into matrix and details are given in Figs. 4.1 and 4.2. Further, fineness of the fly ash plays an important role in the mix. Preliminary testing was based on un-processed type of fly ash which resulted into poor resulted into non-workable concrete and further required strength could not be achieved, therefore processed fly ash as per details 3.2.1 was used.

3.3.5 Mixing and Casting Procedure

Mixing procedure plays a vital role in the production of geopolymer concrete in order to achieve desired properties in fresh and hardened state. For this reason, a set order as had been suggested by M.T. Junaid [35] was followed in the concrete mixing during this study.

Cube, cylinder and beam moulds were first well-oiled for pouring concrete as shown in Fig. 3.15. For the use of geopolymer concrete, waste oil was used as the mould release, as the usual grease would not work the same as with cement based concretes. The alkaline solutions consisting of sodium hydroxide and sodium silicate were combined 24 hours prior to producing geopolymer concrete. The mixing procedure for geopolymer concrete was similar to that of conventional concrete. All dry aggregates, alumino-silicate materials (FA or RHA or GGBS) along with alccofine were first added to the pan mixer and mixed for five minutes for proper mixing. After this dry mixing, the alkaline solution and extra water with superplasticiser were then added gradually and mixed for a further five minutes, or, until an adequately combined mixture was formed.

The moulds were then filled on vibrating table in three layers for proper compaction. Each mould being topped up as the vibration caused the elimination of any air voids. Upon completion of the geopolymer concrete placement, moulds were moved from the table and kept at room temperature in the laboratory. Cubes were kept for a rest period of 24 hours so that concrete hardened properly and the specimens could be demoulded without any damage.

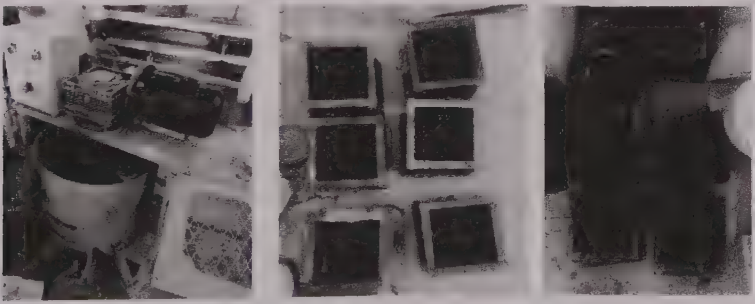


Fig. 3.15: Photograph of pan mixture and prepared moulds

3.3.6 Curing of Specimens

Ambient and heat, two types of curing conditions were adopted during this study. To prevent the geopolymer concrete specimens from excessive evaporation, the samples were wrapped with the plastic film during curing at elevated temperature in an oven. Plastic film suitable upto 250°C temperature was used to cover the specimens [102]. However, ambiently cured specimens were not covered with plastic film.

Preliminary tests also revealed that geopolymer concrete prepared by incorporating alccofine set well even at room temperature. Rest period of 24 hours was given to all the prepared specimens before heat curing. The term 'Rest Period' was coined to indicate the time taken from the completion of casting of test specimens to the start of curing at an elevated temperature. The specimens were cured at ambient as well as heat curing (60°C and 90°C) as shown in Fig. 3.16. As had been discussed in the literature, after 90°C the microstructure become porous, so curing temperature was limited to 90°C.

Fig. 3.16: Photograph of heat curing and ambient curing

3.3.7 Specimens Details

Five cubes, cylinders and beams each of FA, RHA and GGBS were cast for each designated mix in the Tables 3.12 to 3.14. For compressive, split tensile strengths 150mm standard size cubes, for flexural strength beams of size 100x100x500mm standard size beams and for stress-strain curves and modulus of elasticity 150x300mm standard size cylinders were prepared. Cubes and beams were tested after 3, 7 and 28days. Cylinders were tested at the age of 28 days. Further details of the specimens are given in Tables 3.12 to 3.14.

3.4 TESTING METHODS

Workability is the key property to study the fresh properties of the concrete. Structural performance of the concrete mainly depends upon its strength in compression, split tensile and flexure so it is essential to carry out tests to determine the fresh and hardened properties. The following tests have been carried out on concrete and summarized as below:

1. Slump test (ST)
2. Compaction factor (CF)
3. Compressive strength (CS)
4. Flexural strength (FS)
5. Split tensile strength (STS)

3.4.1 Slump test

Slump test is the most common test to evaluate the workability of a fresh concrete in worldwide. Although the slump test does not measure the workability of concrete [69], it is useful to obtain the difference in the consistency of fresh concrete [141] and in detecting variations in the uniformity of concrete mix [69]. Variation in slump indicates that some changes occurred in the batching system or mixing system. The water content in concrete is the most obvious cause, as other factors such as aggregate grading and particle shape may also vary the slump. After the concrete was fully mixed, the fresh concrete was undertaken for use in the slump test (Fig. 3.17). The test procedure was carried out accordance with IS specifications.

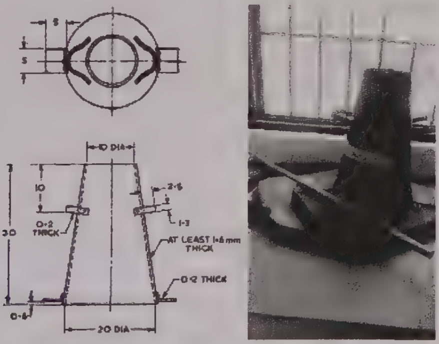


Fig. 3.17: Photograph of slump test mould

3.4.2 Compaction factor test

Compaction factor of fresh concrete is done to determine the workability of fresh concrete by compaction factor test as per IS: 1199 – 1959. The apparatus used was Compaction factor apparatus as is shown in Fig. 3.18.

Fig. 3.18: Experimental setup for compaction factor

Procedure to determine workability of fresh concrete by compaction factor test.

i) The sample of concrete was placed in the upper hopper up to the brim.

ii) The trap-door was opened so that the concrete falls into the lower hopper.

iii) The trap-door of the lower hopper was opened and the concrete was allowed to fall into the cylinder.

iv) The excess concrete remaining above the top level of the cylinder was then cut off with the help of plane blades.

v) The concrete in the cylinder was weighed. This is known as weight of partially compacted concrete.

vi) The cylinder was filled with a fresh sample of concrete in five layers and vibrated to obtain full compaction. The concrete in the cylinder is weighed again. This weight is known as the weight of fully compacted concrete.

Compaction factor = (Weight of partially compacted concrete) / (Weight of fully compacted concrete)

3.4.3 Compressive strength test

The compressive strength of the concrete is very important property as it decides the other properties like tension and flexure. Fig. 3.19 show the experimental set up detail for compressive strength test. Compressive strength test was carried out on 150 mm size standard cubes with compression testing machine of 2000KN capacity as per IS 516:1959 [142]. The procedure used to test the specimens was as follows:

- The specimen and surface of the testing machine was cleaned.
- The specimen was placed at the centre of the compression testing machine and load was applied continuously, uniformly and without shocks and the rate of loading was 14 N/mm^2 (140Kg/cm^2) / minute i.e. at constant rate of stress.
- The load was increased until the specimen failed.
- The maximum load taken by each specimen during the test was recorded.
- The compressive strength was found after 3, 7 and 28 days.

Fig. 3.19: Experimental setup for testing of cube specimen

The compressive strength of the cubes was calculated using the following equation:

$f_c = 1000 \times P / A$...........(3.1)

Where, f_c = compressive strength in MPa

P = maximum applied force in kN

A = area of the cube in mm^2

Cylinder were also tested for their compressive strength and to study stress strain behaviour under compression in the open loop Universal compression testing machine in the laboratory. The details of the test and results is as discussed in the Chapter 5.

3.4.4 Flexural strength test

Flexural strength of concrete has been determined by applying the failure load on prismatic specimen of size 100 mm x 100 mm x 500 mm, using the flexural testing machine of 50KN capacity as per IS 516:1959 [142]. The machine used for the testing is shown in the Fig. 3.20.

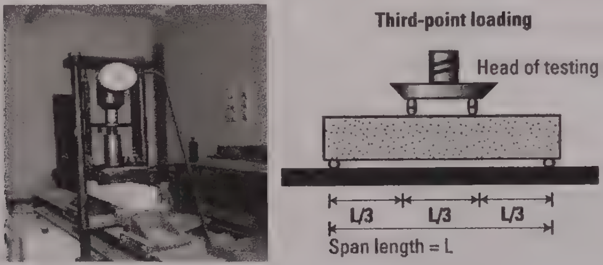


Fig. 3.20: Experimental setup for testing of beam specimen

The flexural strength of the specimen can be expressed as the modulus of rupture f_t.

Modulus of rupture is calculated from following expression:

$$f_t = \frac{pl}{bd^2}$$

When ‘a’ is greater than 133 mm for a 100mm specimen, or

$$f_t = \frac{3pa}{bd^2}$$

If when 'a' is less than 133 mm but greater than 110 mm for a 100mm specimen.

Where

a = the distance between the line of fracture and the nearer support, measured on the center line of the tensile side of the specimen

b = measured width in mm of the specimen,

d = measured depth in mm of the specimen at the point of failure,

l = length in mm of the span on which the specimen was supported,

p = maximum load in N applied to the specimen

If 'a' is less than 110 mm for a 100mm specimen, the results of the test shall be discarded.

3.4.5 Split tensile strength test

The split tensile test is easy to perform and gave more accurate results as compared to the other tensile test such as direct tensile test. The Split tensile test is well known indirect tests used for determining the tensile strength of concrete sometimes referred as the split tensile strength of concrete. The test can be performed on 150mm size cubes by split either (i) along its middle parallel to the edges by applying two opposite compressive forces through 15mm^2 bars of sufficient length, or (ii) along one of the diagonal planes by applying compressive forces along two opposite edges as shown in Fig. 3.21. In the case of side split of these cubes, the tensile strength is determined from 0.642P/S^2 and in diagonal split it is determined from 0.5187 P/S^2, where P is the load at failure and S is the side of the cube. Fig. 3.22 show the failure pattern of the cube.

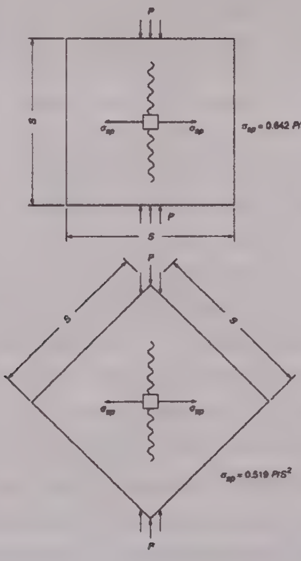


Fig. 3.21: Split strength from testing of cubes

Advantages of the split tensile test for determining the tensile strength are as follows

1. The test is simple to perform and give more uniform results than other tension tests.
2. The strength determined is closer to the actual tensile strength of the concrete than that given by the modulus of rupture test.
3. The same moulds can be used for casting specimens for both compression and tension tests.

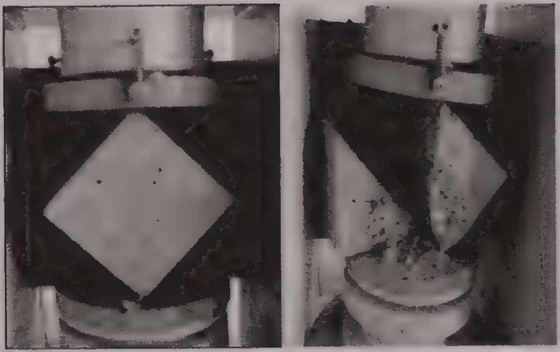


Fig. 3.22: Split tensile test on concrete cubes

3.5 METHODOLOGY

As discussed in the Chapter 2, efforts have been made by the different researchers in formulating the GPC mix design method by adopting the established methods for conventional concrete. In conventional concrete, OPC is the main ingredient among others which imparts binding to the aggregates, provides strength and required workability by forming the hydration products such as C-S-H, C-A-H, ettringite, etc. The required strength and workability parameters are governed by water to cement ratios. In geopolymer concrete, aggregates play the same role as in conventional concrete but other ingredients of the GPC had not been linked to any hydration process. Therefore, it is very difficult to achieve required workability and compressive strength in GPC in 28days at ambient temperature as polymerisation requires high temperature. However, mix design method established by Junaid et al. [35] gave an opportunity to target the required parameters (compressive strength and workability) by using design-aids with certain limitations like, heat curing and only fly ash as a binder was used. Further, workability bands provided in that study did not give any indication related to the slump measurement and again results of the study were not valid for normal curing at room temperature. Therefore, in order to overcome these limitations, a new material alccofine have been proposed as discussed in section 3.3.4, which was rich in lime and gave the combined effect of hydration and polymerisation into the matrix when pozzolanic materials rich in alumino-silicate such as FA, RHA and GGBS were used. The percentage of alccofine into the matrix plays an important role so, preliminary testing was done with different percentages of alccofine and after that a fix amount 10% of alumino-silicate material was fixed for developing the design-aids. Further, controlling ratios in the geopolymer concrete such as water to geopolymer solids (W/GPS), alkaline liquid to alumino-silicate binder, effects of curing etc. have been studied critically and by fixing the parameters as per the details in Table 3.12 to 3.14. The objective of developing design-aids was to determine the mix proportions in terms of quantum of alumino-silicate material, molarity of NaOH, curing temperature, age of curing for a given compressive strength and workability. The proposed method has been further validated with the help of the examples in the end, by targeting strength and workability. The results have also been analysed by using XRD and SEM studies as discussed in 3.5.1.3 and 3.5.1.4.

As geopolymer is the new class of construction material and if the required strength could be achieved after 28days when ambient curing adopted it can be used on large scale in cast-in-situ and precast industry. Therefore, structural properties such as compressive, split tensile, flexural strengths, stress-strain behaviour and modulus of elasticity were also studied for the mixes tabulated in the Table 3.12 to 3.14. The results of the structural properties have been discussed in the next chapter.

3.5.1 MIXTURE PROPORTIONS FOR PRELIMINARY LABORATORY WORK

The mix design of geopolymer concrete with fly ash was done in reference to proposed mix design by Junaid et al. [35] for the different proportion of fly ash. Similar to that of conventional concrete, coarse and fine aggregates were taken approximately 75-77% by mass of the entire mixture. The concentration of NaOH solution was 16 M so as to achieve better compressive strength [51]. 2% Naphthalene Sulphonate based superplasticizer was used to improve the workability of fresh geopolymer mix. Higher W/GPS ratio improves the workability but reduces the compressive strength. Therefore, a fixed W/GPS ratio of 0.27 was used to achieve higher compressive strength. Alkaline liquid to fly ash (AL/FA) ratio was kept 0.38, 0.42 and 0.46, respectively, for mix designated M1A0, M2A0, and M3A0, respectively. Mix M1A0, M2A0, and M3A0 contain fly ash contented 350 kg, 370 kg and 400 kg per cubic meter of geopolymer mix. The details of mix proportions of geopolymer concrete mixes is given in Table 3.11. GPC mixes with different amount of Alccofine 1203 (0%, 5%, and 10%) were also cast to analyse its effect on workability and compressive strength of geopolymer concrete.

Table 3.11: Mix proportion of geopolymer concrete mixes.

Mix No./ Designation	Quantity of ingredients (kg/m^3)								
	Coarse Aggregates			Fine Aggregates	Fly Ash	Alccofine (% age of fly ash)	NaOH	Na_2SiO_3	Extra Water
	14 mm	10 mm	7 mm						
M1A0/M1A0P	614	460	269	575	350	0.0	38	95	36.02
M1A0UP	614	460	269	575	350	0.0	38	95	36.02
M1A5	614	460	269	575	350	5	38	95	36.02
M1A10	614	460	269	575	350	10	38	95	36.02
M2A0/M2A0P	600	450	260	565	370	0.0	44.4	111	31.58
M2A0UP	600	450	260	565	370	0.0	44.4	111	31.58
M2A5	600	450	260	565	370	5	44.4	111	31.58
M2A10	600	450	260	565	370	10	44.4	111	31.58
M3A0/M3A0P	565	445	255	540	400	0.0	52.58	131.45	27.07
M3A0UP	565	445	255	540	400	0.0	52.58	131.45	27.07
M3A5	565	445	255	540	400	5	52.58	131.45	27.07
M3A10	565	445	255	540	400	10	52.58	131.45	27.07

M1A0P, M1A0UP (Mix 1 , Alccofine 0%, Processed fly ash , unprocessed fly ash, respectively)

3.5.1.1 WORKABILITY

Workability of GPC mixes was studied using slump cone test and is presented in Fig. 3.23. The fresh geopolymer concrete mixes were observed highly harsh and particularly in the case of GPC with unprocessed fly ash which produced slump less concrete. Workability of fresh geopolymer mix was significantly improved by using 2% Naphthalene Sulphonate based superplasticizer as had also been observed by [143]. It was observed that the workability of geopolymer concrete was very low in case of mixture without alccofine.

3.5.1.1.1 Effect of type and content of fly ash

Geopolymer mixture prepared with unprocessed fly ash resulted into slump less concrete i.e. the slump obtained was zero. Fig. 3.23 shows that GPC mixes made with processed type of fly ash provided a measurable slump which improved significantly on increasing the fly ash content. The increment in slump value was observed from 20mm to 120mm on increasing fly ash content from 350kg/m^3 to 400kg/m^3.

The zero-slump obtained in case of unprocessed fly ash was due to the presence of unburned carbon particles which makes fly ash hygroscopic. Therefore, the noticed increase in slump with processed fly ash might be with the increase in fine spherical particles and with the increase in fly ash content.

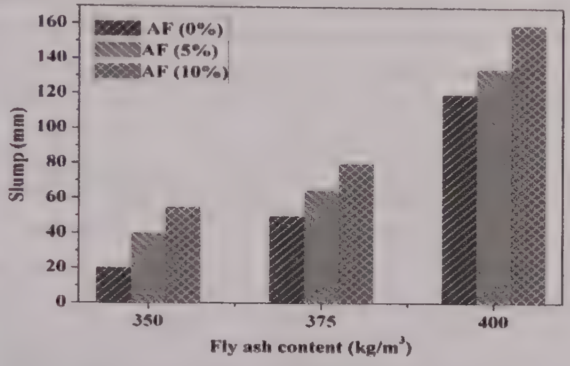


Fig. 3.23: Workability (slump) of fly ash based geopolymer concrete with different content of alccofines from 0% to 10%. (FA- Fly ash, AF-Alccofine)

3.5.1.1.2 Effect of alccofine content

Geopolymer concrete mix containing alccofine resulted into good workable concrete showing a significant increase in workability on increasing the alccofine content as shown in Fig. 3.23. A collapse slump was observed with 10% alccofine content due to its microfined structure with fineness more than 12000 cm^2/gm [135]. Further, increase in slump was about 200% for the mixes with 10% alccofine with minimum fly ash content to that of maximum fly ash content without alccofine. Also, slump values increased by 29-30% and 33-34% when alccofine amount changed from 0 to 5% and 5 to 10% with same fly ash content. Alccofine due to its controlled higher fineness and spherical particle size resulted into increased ball bearing effect which enhanced the workability of GPC.

3.5.1.2 COMPRESSIVE STRENGTH

The compressive strength of GPC cast with different type of fly ash and alccofine contents along with method of curing has been discussed in the following paragraphs:

3.5.1.2.1 Effect of type of fly ash and method of curing

Influence of type of fly ash on compressive strength of geopolymer concrete (GPC) has been illustrated in Figs. 3.24 and 3.25. It shows that the compressive strength achieved by GPC has improved in case of processed fly ash in ambient as well as in heat cured conditions. The compressive strength of the GPC (Mix M1A0UP) using unprocessed fly ash increased from

2MPa to 6MPa and 4MPa to 12MPa with the increase in age from 3days and 28days for ambient and heat cured specimens, respectively. However, the compressive strength increased from 5MPa to 12MPa and 10MPa to 20MPa when GPC (Mix M1A0P) based on processed fly ash without alccofine was tested at the ages of 3days and 28days at ambient and heat curing, respectively.

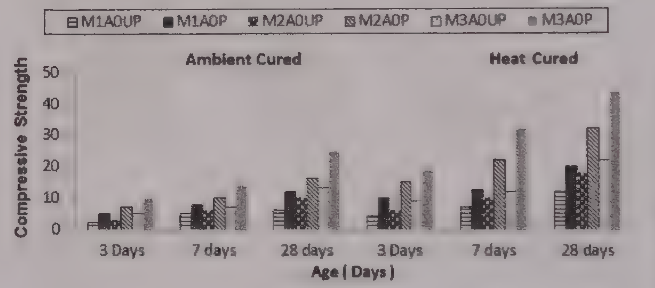


Fig. 3.24: Comparison of compressive strength of processed and unprocessed fly ash based GPC.

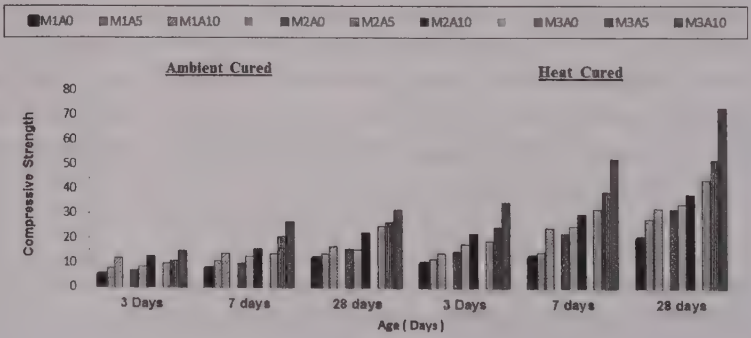


Fig. 3.25: Effects of variation of fly ash content, alccofine, curing type and age of casting on the compressive strength of fly ash based geopolymer concrete.

It is evident from the above graph that GPC prepared with 400kg of processed fly ash at ambient curing can be used up to 20MPa compressive strength. The compressive strength of the sample M3A0P (FA=400kg) further increased when the temperature was raised from ambient to 90^{0}C. The results obtained show nearly 300% to 130% increment in early compressive strength (3 and 7 days) of the heat cured and ambiently cured specimens were compared. This increase in compressive strength at higher temperature had been due to the properties of fly ash concrete.

3.5.1.2.2 Effect of fly ash content

Effect of variation of fly ash content on compressive strength of GPC with processed fly ash is shown in Fig. 3.25. It was observed that early ages (3 and 7 days) compressive strength of GPC prepared using processed fly ash increased from 5 MPa to 7 MPa and 10 MPa, 7.5MPa to 10MPa and 16 MPa as well as 28 days compressive strength increased from 12 MPa to 16 MPa and 25 MPa in case of ambient curing on varying the fly ash content from 350 kg to 370 kg and 400 kg, respectively. It is as such obvious that the processed fly ash geopolymer concrete could achieve minimum required compressive strength used for general construction purpose. Whereas the highest compressive strength obtained at 400kg/m^3 unprocessed fly ash content at ambient temperature was 13MPa which was less than 20MPa, the required characteristic strength of M20 (BIS 456) [134].

Increased fly ash content attributes to the increased quantity of binder material as well as the development of denser concrete improving the compressive strength parameter. Geopolymer concrete based at processed fly ash results into better concrete in comparison to unprocessed fly ash due to better fineness and controlled chemical composition.

3.5.1.2.3 Effect of alccofine content

Effect of alccofine content on compressive strength of various mixes is shown in Fig. 3.25. It was observed that early ages (3 and 7 days) compressive strength of processed fly ash based GPC (M1A5 and M1A10) increased in the range of 20% to 45% whereas up to 62% at the age of 28 days when heat curing adopted. A similar pattern was observed in the case of higher fly ash content. Therefore, it shows that the addition of alccofine improves the early age as well as ultimate compressive strength. A compressive strength of GPC of 35 MPa and 15 MPa can be developed at the age of 3 days using 10% alccofine using heat and ambient curing, which resulted into 73 MPa and 32 MPa at the age of 28 days, respectively. However, it was also observed that at higher fly ash content, the percentage increase in compressive strength is relatively less in comparison to GPC mix with lower fly ash content.

It can, therefore, be seen that geopolymer concrete with alccofine can achieve target compressive strength for M25 even when the specimens were ambiently cured. Literature, however, has insisted that heat curing is essential for geopolymer concrete to meet minimum required compressive strength. There remains no doubt that heat curing makes alccofine more efficient at higher fly ash content and the same specimens could reach 73 MPa. This indicates that geopolymer concrete with alccofine can be very useful for general purpose construction and for precast industries.

If the properties of fly ash and alccofine are compared, it is noticeable that alccofine has higher fineness and is rich in alumina content. Therefore, the hydration is more effective in the presence of alccofine and polymerisation in the presence of rich silica material as fly ash. Moreover, ultra-high fineness of alccofine subsequently may have plugged the microspores enhancing the compressive strength of geopolymer concrete.

3.5.1.3 X-RAY DIFFRACTION (XRD) STUDIES

Fig. 3.26 show X-ray diffraction curves for geopolymer concrete with and without alccofine. The same Fig. also shows the curves for alccofine and fly ash. A comparison which immediately clarifies that polymerization has transformed amorphous material into crystal material and more significantly in the presence of alccofine. The intensity of quartz at 2θ= 26.60 has increased with increase in alccofine content. Further, the peak of C-S-H at 2θ= 40.30 have decreased with the increase in alccofine content. This can also be concluded [144] that water is not structurally bound in geopolymer concrete and at higher alccofine content water molecules in the form of hydrates and hydroxides decreased. However, many peaks at 2θ= 54.0, 38.50 and 67.70 of calcium magnesium silicates were observed in geopolymer concrete. All these reasons might have resulted in better compressive strength in the presence of alccofines. Previous studies on the fly ash based geopolymer concrete show mullite, quartz, and nepheline, which is associated with NASH, formed by polymerisation reactions [145]. However, the inclusion of alccofine in the GPC matrix forms CSH in addition to other compounds and contributes to achieving the higher strength at ambient and heat curing. Also, with the increase in alccofine percentage high peaks of the quartz and mullite were observed. As strength increases with the increase in the percentage of alccofine [136], it can be assumed that at higher alccofine percentage dense matrix will form and higher peaks of quartz could be observed. The XRD results reveal that the prepared sample present in a crystalline form which also clearly observed in SEM image.

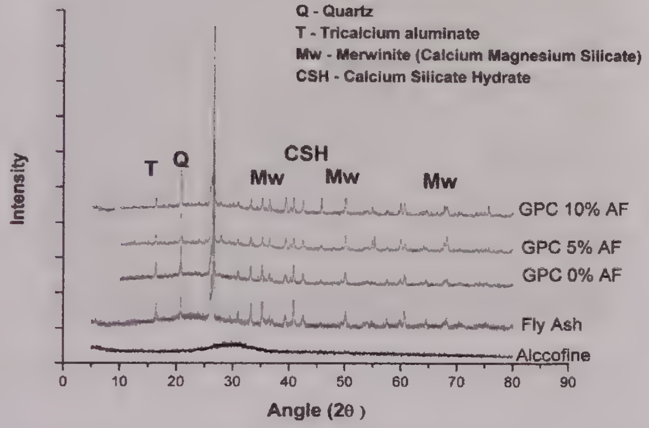


Fig. 3.26: XRD curves for Geopolymer Concrete with and without alccofines.

3.5.1.4 SCANNING ELECTRON MICROSCOPE (SEM) STUDIES

SEM analyses were conducted on prepared specimens, in order to identify the reactants of fly ash and to verify the internal microstructure. SEM microscopy of the samples was measured using a Hitachi S-3000N. The size of the samples was approximately 12 x 10 mm. The working distance and the voltage for this analysis were approximately 10 to 25 mm and 7 kV, respectively. However, the images were inspected at 250X magnification. Figs. 3.27 to 3.29 show the microstructures of geopolymer concrete with and without alccofine. Geopolymer concrete without alccofines wherein aluminates and hydroxides as white precipitates can be observed. However, the microstructure of geopolymer concrete with alccofine in Fig. 3.28 and Fig. 3.29 is entirely different. Fig. 3.28 shows the presence of C-S-H and polymerization in the form of white precipitates as has been confirmed in XRD studies. Spherical cavities of fly-ash particles are minimum in Fig. 3.29. Therefore, the increase in compressive strength with 10% alccofine can be due to better polymerization and hydration. Better polymerization further resulted in better crystallization and densely packed microstructure.

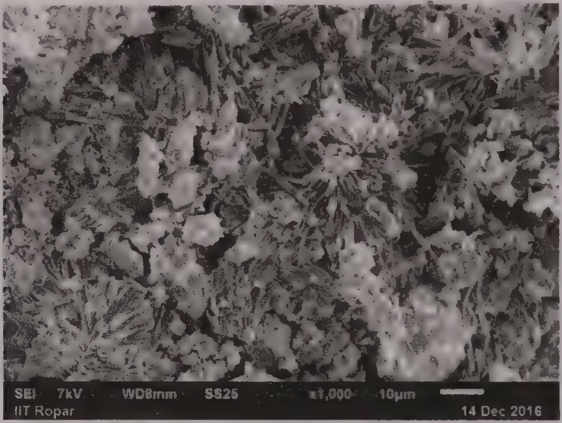


Fig. 3.27: SEM micrograph of geopolymer concrete without alccofines.

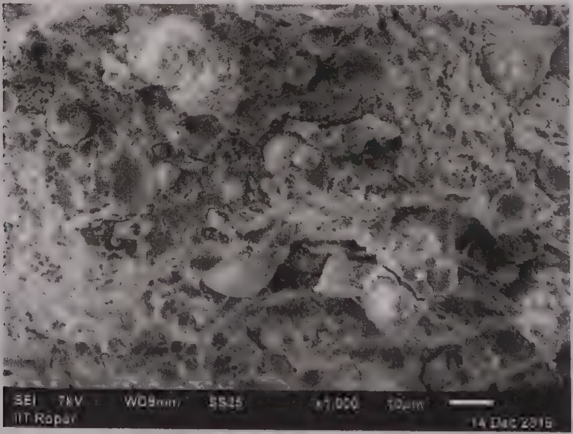


Fig. 3.28: SEM micrograph of geopolymer concrete with 5% alccofines.

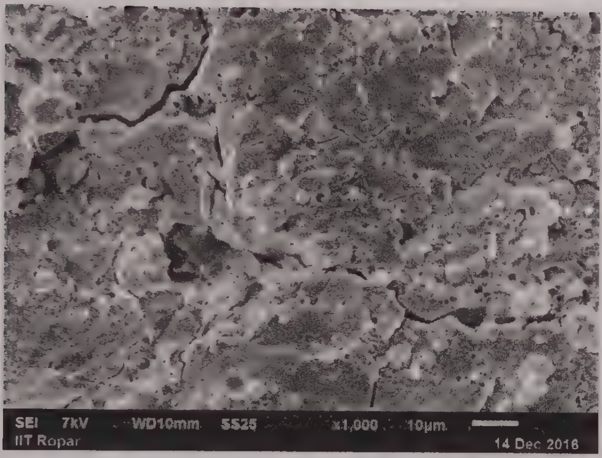


Fig. 3.29: SEM micrograph of geopolymer concrete with 10% alccofines.

Based on the above results and discussions presented, following conclusions can be derived upon:

- Unprocessed fly ash doesn't gain any desirable mechanical properties.
- The presence of alccofine produces better workable concrete with processed and unprocessed fly ash geopolymer concrete.
- Minimum required compressive strength for general construction purpose can be achieved with alccofine even at room temperature using processed fly ash.
- The increase in compressive strength was significant at 90^0C in the presence of alccofine and perhaps provides an opportunity for the most economical and sustainable way to achieve higher compressive strength.
- XRD and SEM study points out that on the addition of alccofine, amorphous material changes into the crystalline material which is responsible for the improved compressive strength of GPC.

3.5.2 MIXTURE PROPORTIONS FOR DESIGN AIDS

Based on the up-to date literature related to past research on geopolymer concrete prepared using different waste materials and the experience gained during the preliminary experimental work done using the different alccofine contents (refer clause 3.4), the following ranges were selected for the constituents of the mixtures used in further studies.

- Low calcium class F dry fly ash as given in Section 3.2.1.
- Dry rice husk ash rich in silica as given in Section 3.2.2.
- Dry GGBS as shown in Section 3.2.3.
- Alkaline liquid as given in Section 3.2.2.
- Ratio of sodium silicate solution-to-sodium hydroxide solution, by mass 2.5. This ratio was fixed at 2.5 for all the mixtures designated in Tables 3.12 to 3.14 as sodium silicate solution is considerably cheaper than the sodium hydroxide solution.
- Molarity of sodium hydroxide (NaOH) solution- 8M, 12M and 16M. These ranges were selected as GPC strength very much depends on the molarity.
- Ratios of activator solution-to-fly ash and water to geopolymer binder (by weight), 0.45 and 0.27, respectively, were fixed in order to achieve the better workability.
- Alccofine amount was fixed as 10% of the alumino-silicate material by weight as required workability and compressive strength could be achieved.
- Amount of alumino-silicate materials (FA, RHA and GGBS) was taken as 350, 375 and 400kg/m^3. Different quantities were selected to cover a wide practicable range of compressive strength which further can be utilised to develop a mix design procedure.

The mixture proportions used in this study are as shown in Tables 3.12 to 3.14, respectively, for fly ash, RHA and GGBS based geopolymer concrete. Mixes M1FAGC to M9FAGC were prepared in anticipating of it being reference mix by which other mixes (M10FAGC to M27FAGC) were prepared by changing the curing temperature (to be in tables part).

Table 3.12: Mixture proportions used for fly ash based geopolymer concrete

Mixture	Fine Aggregate [Kg/m^3]	Coarse Aggregate [Kg/m^3]	Fly Ash [Kg/m^3]	Molarity: NaOH [M]	Total Alkaline Solution [Kg/m^3]	NaOH [Kg/m^3]	Na_2SiO_3	Extra water [Kg/m^3]	Alccofine [Kg/m^3]	Super plasticizer [Kg/m^3]	Curing Temperature (°C) / Rest Period (hours.)
M1FAGC	533	1243	350	8	157.5	45.00	112.5	26	35.0	7.0	27 / 24
M2FAGC	521	1215	375	8	168.7	48.21	120.5	28	37.5	7.5	27 / 24
M3FAGC	508	1186	400	8	180.0	51.42	128.5	28	40.0	8.0	27 / 24
M4FAGC	531	1239	350	12	157.5	45.00	112.5	30	35.0	7.0	27 / 24
M5FAGC	519	1210	375	12	168.7	48.21	120.5	30	37.5	7.5	27 / 24
M6FAGC	506	1181	400	12	180.0	51.42	128.5	32	40.0	8.0	27 / 24
M7FAGC	530	1236	350	16	157.5	45.00	112.5	32	35.0	7.0	27 / 24
M8FAGC	517	1207	375	16	168.7	48.21	120.5	34	37.5	7.5	27 / 24
M9FAGC	505	1178	400	16	180.0	51.42	128.5	36	40.0	8.0	27 / 24
M10FAGC	533	1243	533	8	157.5	45.00	112.5	26	35.0	7.0	27 / 24
M11FAGC	521	1215	521	8	168.7	48.21	120.5	28	37.5	7.5	60 / 24
M12FAGC	508	1186	508	8	180.0	51.42	128.5	28	40.0	8.0	60 / 24
M13FAGC	531	1239	531	12	157.5	45.00	112.5	30	35.0	7.0	60 / 24
M14FAGC	519	1210	519	12	168.7	48.21	120.5	30	37.5	7.5	60 / 24
M15FAGC	506	1181	506	12	180.0	51.42	128.5	32	40.0	8.0	60 / 24
M16FAGC	530	1236	530	16	157.5	45.00	112.5	32	35.0	7.0	60 / 24
M17FAGC	517	1207	517	16	168.7	48.21	120.5	34	37.5	7.5	60 / 24
M18FAGC	505	1178	505	16	180.0	51.42	128.5	36	40.0	8.0	60 / 24
M19FAGC	533	1243	350	8	157.5	45.00	112.5	26	35.0	7.0	90 / 24
M20FAGC	521	1215	375	8	168.7	48.21	120.5	28	37.5	7.5	90 / 24

M21FAGC	508	1186	400	8	180.0	51.42	128.5	28	40.0	8.0	90 / 24
M22FAGC	531	1239	350	12	157.5	45.00	112.5	30	35.0	7.0	90 / 24
M23FAGC	519	1210	375	12	168.7	48.21	120.5	30	37.5	7.5	90 / 24
M24FAGC	506	1181	400	12	180.0	51.42	128.5	32	40.0	8.0	90 / 24
M25FAGC	530	1236	350	16	157.5	45.00	112.5	32	35.0	7.0	90 / 24
M26FAGC	517	1207	375	16	168.7	48.21	120.5	34	37.5	7.5	90 / 24
M27FAGC	505	1178	400	16	180.0	51.42	128.5	36	40.0	8.0	90 / 24

Table 3.13: Mixture proportions used for rice husk ash based geopolymer concrete

Mixture	Fine Aggregate [Kg/m^3]	Coarse Aggregate [Kg/m^3]	Rice Husk Ash [Kg/m^3]	Molarity: NaOH [M]	Total Alkaline Solution [Kg/m^3]	NaOH [Kg/m^3]	Na_2SiO_3	Extra water [Kg/m^3]	Alccofine [Kg/m^3]	Super plasticizer [Kg/m^3]	Curing Temperature (^{o}C) / Rest Period (hours.)
M1RHAGC	533	1243	350	8	157.5	45.00	112.5	26	35.0	7.0	27 / 24
M2RHAGC	521	1215	375	8	168.7	48.21	120.5	28	37.5	7.5	27 / 24
M3RHAGC	508	1186	400	8	180.0	51.42	128.5	28	40.0	8.0	27 / 24
M4RHAGC	531	1239	350	12	157.5	45.00	112.5	30	35.0	7.0	27 / 24
M5RHAGC	519	1210	375	12	168.7	48.21	120.5	30	37.5	7.5	27 / 24
M6RHAGC	506	1181	400	12	180.0	51.42	128.5	32	40.0	8.0	27 / 24
M7RHAGC	530	1236	350	16	157.5	45.00	112.5	32	35.0	7.0	27 / 24
M8RHAGC	517	1207	375	16	168.7	48.21	120.5	34	37.5	7.5	27 / 24
M9RHAGC	505	1178	400	16	180.0	51.42	128.5	36	40.0	8.0	27 / 24
M10RHAGC	533	1243	350	8	157.5	45.00	112.5	26	35.0	7.0	27 / 24
M11RHAGC	521	1215	375	8	168.7	48.21	120.5	28	37.5	7.5	60 / 24
M12RHAGC	508	1186	400	8	180.0	51.42	128.5	28	40.0	8.0	60 / 24
M13RHAGC	531	1239	350	12	157.5	45.00	112.5	30	35.0	7.0	60 / 24
M14RHAGC	519	1210	375	12	168.7	48.21	120.5	30	37.5	7.5	60 / 24
M15RHAGC	506	1181	400	12	180.0	51.42	128.5	32	40.0	8.0	60 / 24
M16RHAGC	530	1236	350	16	157.5	45.00	112.5	32	35.0	7.0	60 / 24
M17RHAGC	517	1207	375	16	168.7	48.21	120.5	34	37.5	7.5	60 / 24
M18RHAGC	505	1178	400	16	180.0	51.42	128.5	36	40.0	8.0	60 / 24

M19RHAGC	533	1243	350	8	157.5	45.00	112.5	26	35.0	7.0	90 / 24
M20RHAGC	521	1215	375	8	168.7	48.21	120.5	28	37.5	7.5	90 / 24
M21RHAGC	508	1186	400	8	180.0	51.42	128.5	28	40.0	8.0	90 / 24
M22RHAGC	531	1239	350	12	157.5	45.00	112.5	30	35.0	7.0	90 / 24
M23RHAGC	519	1210	375	12	168.7	48.21	120.5	30	37.5	7.5	90 / 24
M24RHAGC	506	1181	400	12	180.0	51.42	128.5	32	40.0	8.0	90 / 24
M25RHAGC	530	1236	350	16	157.5	45.00	112.5	32	35.0	7.0	90 / 24
M26RHAGC	517	1207	375	16	168.7	48.21	120.5	34	37.5	7.5	90 / 24
M27RHAGC	505	1178	400	16	180.0	51.42	128.5	36	40.0	8.0	90 / 24

Table 3.14: Mixture proportions used for GGBS based geopolymer concrete

Mixture	Fine Aggregate [Kg/m^3]	Coarse Aggregate [Kg/m^3]	GGBS [Kg/m^3]	Molarity: NaOH [M]	Total Alkaline Solution [Kg/m^3]	NaOH [Kg/m^3]	Na_2SiO_3	Extra water [Kg/m^3]	Super plasticizer [Kg/m^3]	Curing Temperature (°C) / Rest Period (hours.)
M1GGBSGC	533	1243	350	8	157.5	45.00	112.5	15	7.0	27 / 24
M2GGBSGC	521	1215	375	8	168.7	48.21	120.5	15	7.5	27 / 24
M3GGBSGC	508	1186	400	8	180.0	51.42	128.5	15	8.0	27 / 24
M4GGBSGC	531	1239	350	12	157.5	45.00	112.5	20	7.0	27 / 24
M5GGBSGC	519	1210	375	12	168.7	48.21	120.5	20	7.5	27 / 24
M6GGBSGC	506	1181	400	12	180.0	51.42	128.5	22	8.0	27 / 24
M7GGBSGC	530	1236	350	16	157.5	45.00	112.5	27	7.0	27 / 24
M8GGBSGC	517	1207	375	16	168.7	48.21	120.5	27	7.5	27 / 24
M9GGBSGC	505	1178	400	16	180.0	51.42	128.5	27	8.0	27 / 24
M10GGBSGC	533	1243	350	8	157.5	45.00	112.5	15	7.0	27 / 24
M11GGBSGC	521	1215	375	8	168.7	48.21	120.5	15	7.5	60 / 24
M12GGBSGC	508	1186	400	8	180.0	51.42	128.5	15	8.0	60 / 24
M13GGBSGC	531	1239	350	12	157.5	45.00	112.5	20	7.0	60 / 24
M14GGBSGC	519	1210	375	12	168.7	48.21	120.5	20	7.5	60 / 24
M15GGBSGC	506	1181	400	12	180.0	51.42	128.5	22	8.0	60 / 24
M16GGBSGC	530	1236	350	16	157.5	45.00	112.5	27	7.0	60 / 24
M17GGBSGC	517	1207	375	16	168.7	48.21	120.5	27	7.5	60 / 24
M18GGBSGC	505	1178	400	16	180.0	51.42	128.5	27	8.0	60 / 24
M19GGBSGC	533	1243	350	8	157.5	45.00	112.5	15	7.0	90 / 24

M20GGBSGC	521	1215	375	8	168.7	48.21	120.5	15	7.5	90 / 24
M21GGBSGC	508	1186	400	8	180.0	51.42	128.5	15	8.0	90 / 24
M22GGBSGC	531	1239	350	12	157.5	45.00	112.5	20	7.0	90 / 24
M23GGBSGC	519	1210	375	12	168.7	48.21	120.5	20	7.5	90 / 24
M24GGBSGC	506	1181	400	12	180.0	51.42	128.5	22	8.0	90 / 24
M25GGBSGC	530	1236	350	16	157.5	45.00	112.5	27	7.0	90 / 24
M26GGBSGC	517	1207	375	16	168.7	48.21	120.5	27	7.5	90 / 24
M27GGBSGC	505	1178	400	16	180.0	51.42	128.5	27	8.0	90 / 24

Summary:- This Chapter has provided the details of materials properties, casting of geopolymer test specimens, test procedures, details of preliminary laboratory work with results and methodology to achieve the objectives as appeared in Chapter 1. The samples were prepared to determine the structural properties like compressive, split tensile and flexural strengths. Cylinders were also cast to study the stress-stain behaviour and Young's modulus of elasticity of the geopolymer concrete. The results obtained during the above experimental program related to the structural properties of geopolymer concrete have been discussed in the Chapter 4 while analysis in Chapter 5.

CHAPTER - 4

RESULTS AND DISCUSSIONS

4.1 INTRODUCTION

This chapter presents the results of the experimental testing undertaken for the understanding of the fresh and hardened properties of the geopolymer concrete based on FA, RHA and GGBS as alumino-silicate material. The fresh properties cover the workability in terms of slump and compaction factor and hardened properties cover compressive, split tensile and flexural strengths.

4.2 WORKABILITY OF THE GEOPOLYMER CONCRETE

4.2.1 Fly Ash Based Geopolymer Concrete

The results of workability obtained for the mixes M1FAGC to M9FAGC, in terms of slump value (mm) and compaction factor are shown in Table 4.1. The test results of the workability are depicted with the help of the Figs. 4.1 and 4.2.

Table 4.1: Slump and compaction factor variation in fly ash based GPC

Mix Designation	Fly ash [Kg/m^3] / Molarity	Slump (mm)	Compaction factor
M1FAGC	350 / 8M	60	0.79
M2FAGC	375 / 8M	110	0.87
M3FAGC	400 / 8M	160	0.95
M4FAGC	350 / 12M	55	0.76
M5FAGC	375 / 12M	105	0.85
M6FAGC	400 / 12M	155	0.93
M7FAGC	350 / 16M	50	0.75
M8FAGC	375 / 16M	100	0.82
M9FAGC	400 / 16M	140	0.90

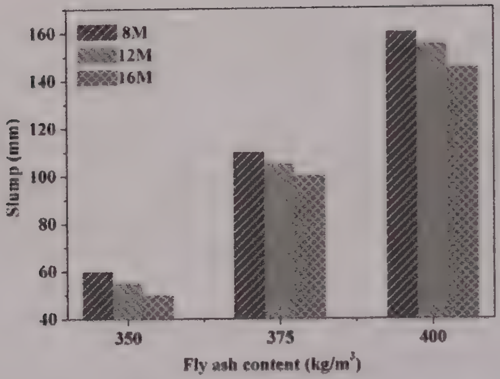


Fig. 4.1: Variation of slump with fly ash content

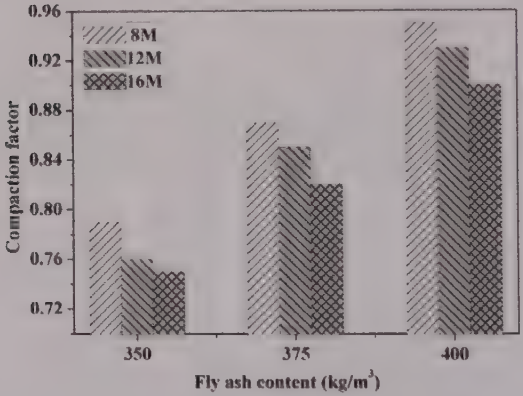


Fig. 4.2: Variation of compaction factor with fly ash content

A measurable slump of 60 mm was noticed in GPC with fly ash of 350 kg/m^3 (M1FAGC). Further, the slump value improved from 60 mm to 110 mm and 160 mm on increasing fly ash content from 350 to 370 (M2FAGC) and 400 (M3FAGC) kg/m^3, respectively, as indicated in Fig. 4.1. Similar trend was also observed in case of compaction factor values for the same mixes as indicated in Fig. 4.2. The increase in the slump might be due to increase in finer content of alccofine and superplasticiser with the increase in fly ash content.

Mixes M3FAGC, M6FAGC and M9FAGC are also compared to study the effect of NaOH concentration on the workability of the geopolymer concrete. Mix M9FAGC exhibited a slightly lower slump (140 mm) than mix M6FAGC (155 mm) and mix M3FAGC exhibited a significantly higher slump (160 mm) than the mix M6FAGC (155 mm) and the slump collapse as shown in Fig. 4.3. Similar trend was observed in compaction factor values for the same mixes. It is clear from the above discussions that NaOH molarity affects the workability. With the increase in molarity the slump and compaction factor values decreased. The use of sodium hydroxide and sodium silicate solution, which are highly viscous than water made the geopolymer concrete more cohesive and stiff than conventional concrete, may be the reason of less workable geopolymer concrete.

The extra water shown in Tables above was determined through the ratio of W-GPS, however, it seems that to achieve higher workability extra water required would be more, which may further lessen the strength.

Fig. 4.3: Fresh geopolymer concrete with collapsed slump (M3FAGC)

4.2.2 Rice Husk Ash Based Geopolymer Concrete

The results of workability obtained for the mixes M1RHAGC to M9RHAGC, in terms of slump value (mm) and compaction factor are shown in Table 4.2. The test results of the workability are depicted with the help of the Figs. 4.4 and 4.5.

Table 4. 2: Slump and compaction factor variation in rice husk ash based GPC

Mix Designation	Rice husk ash [Kg/m^3] / Molarity	Slump (mm)	Compaction factor
M1RHAGC	350 / 8M	50	0.74
M2RHAGC	375 / 8M	95	0.80
M3RHAGC	400 / 8M	145	0.88
M4RHAGC	350 / 12M	45	0.71
M5RHAGC	375 / 12M	85	0.76
M6RHAGC	400 / 12M	135	0.85
M7RHAGC	350 / 16M	40	0.67
M8RHAGC	375 / 16M	75	0.70
M9RHAGC	400 / 16M	120	0.80

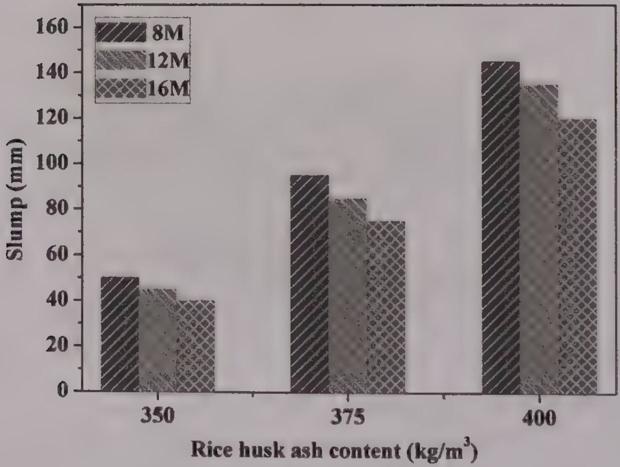


Fig. 4.4: Variation of slump with rice husk ash content

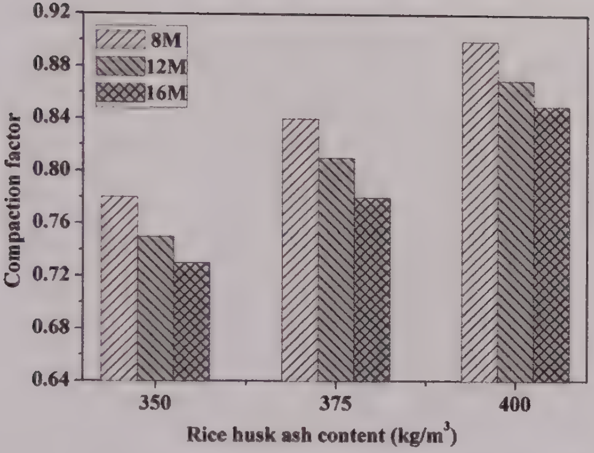


Fig. 4.5: Variation of compaction factor with rice husk ash content

A similar pattern of the slump was obtained in RHA based GPC as in the case of fly ash based GPC but with a lower value increasing from 50 mm to 95 mm & 145 mm when mix M1RHAGC is compared with M2RHAGC & M3RHAGC. The decrease in slump when compared with the fly ash based geopolymer concrete might be due to high water requirements of the rice husk ash [146]. Mix M9RHAGC exhibited a lower value of slump among all the mixes which show that NaOH molarity affected the fresh properties of the RHA based GPC in the same manner when compared it with FA based GPC. It has been observed that alccofine and superplasticizer improved the workability of the RHA based GPC in the similar manner as had been discussed for fly ash.

4.2.3 GGBS Based Geopolymer Concrete (GGBS)

The results of workability obtained for the mixes M1GGBSGC to M9GGBSGC, in terms of slump value (mm) and compaction factor was shown in Table 4.3. The test results of the workability are depicted with the help of the Figs. 4.6 and 4.7.

Table 4. 3: Slump and compaction factor variation in GGBS based GPC

Mix Designation	GGBS [Kg/m^3] / Molarity	Slump (mm)	Compaction factor
M1GGBSGC	350 / 8M	50	0.82
M2GGBSGC	375 / 8M	85	0.89
M3GGBSGC	400 / 8M	140	0.96
M4GGBSGC	350 / 12M	45	0.81
M5GGBSGC	375 / 12M	75	0.87
M6GGBSGC	400 / 12M	125	0.95
M7GGBSGC	350 / 16M	40	0.77
M8GGBSGC	375 / 16M	70	0.86
M9GGBSGC	400 / 16M	110	0.93

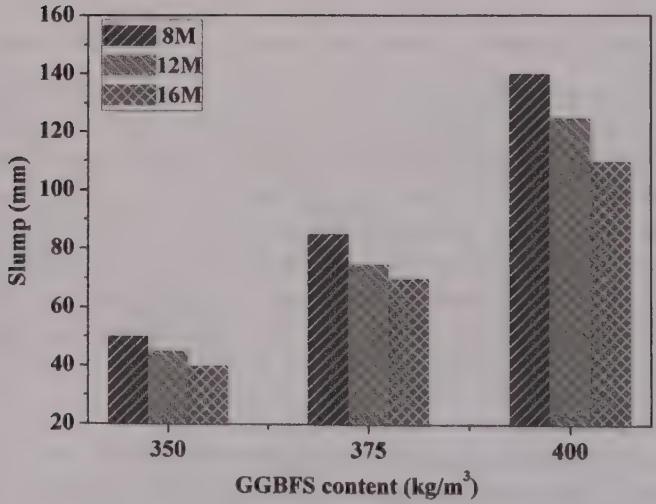


Fig. 4.6: Variation of slump with GGBS content

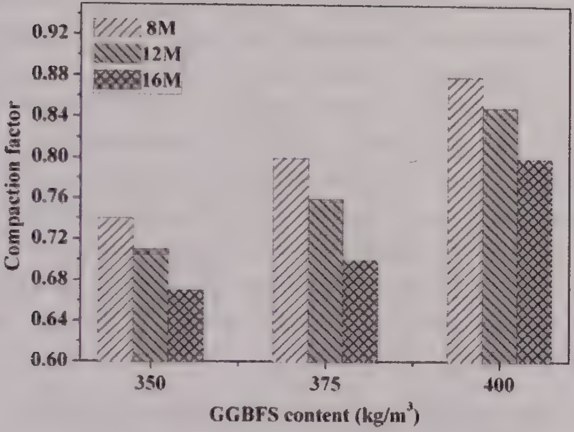


Fig. 4.7: Variation of compaction factor with GGBS content

Geopolymer concrete produced with GGBS has similar pattern of the slump and compaction factor as was obtained in RHA and FA based GPC. The values were however lower. The decrease in slump values when compared with the FA and RHA based geopolymer concrete may be due to its chemical composition of the GGBS which had a highest amount of SiO_2 and CaO. Also, GGBS was finer and had more specific area compared to FA and RHA. This might result in higher water requirement and therefore the workability was minimum. Workability of the GPC decreases with the increase in fineness of the binders, it has also been confirmed by Patankar [123].

It can be concluded from the above results of workability obtained in this study that geopolymer concrete produced with FA / RHA / GGBS as a binder material is workable when it is produced with the alccofine and superplasticiser.

The prominent reason of the poor workability of the geopolymer concrete produced in the literature may be due to the increased fineness of binder material [123]. Further, GPC requires less water as compare to conventional concrete as polymerisation dominates. The variation in the slump value with different type of binders is also because of the variation in their fineness, shape of the particles and chemical composition. Further, the decrease in the slump values with the increase in NaOH molarity may be due to hardening process, as alccofine is rich in lime. Also, the increase in calcium content was not only due to presence of alccofine but also due to its binder sources like fly ash, rice husk ash and GGBS, etc. These binders formed additional nucleation sites within the matrix which increased the rate of solidification and hence increased its hardening [41, 92, 140]. The use of superplasticiser along with alccofine may have resulted in less friction and better viscous material for suspended mass of aggregates which has enhanced the workability of GPC in all types of geopolymer concretes in this study. Further, as the slump test alone is not a good measure of workability, therefore, compaction factor test was done to confirm the applicability of the geopolymer concrete in fresh state. The trend of the compaction factor and slump was very much similar. So, it can be concluded that alccofine can be used in order to produce the homogenous geopolymer concrete which also improves its rheological characteristics including compatibility, mobility and stability.

4.3 STRUCTURAL PROPERTIES OF THE GEOPOLYMER CONCRETE

4.3.1 Fly Ash Based Geopolymer Concrete

The effects of different parameters on the structural properties such as compressive, split tensile and flexural strengths of the fly ash based geopolymer concrete has been discussed below.

4.3.1.1 Compressive strength

The influences of NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine into geopolymer concrete on the compressive strength were studied. The compressive strength was measured after 3, 7 and 28 days. The specimens were cured at 27° C, 60° C and 90° C. The average compressive strength of five specimens is reported in the Table 4.4 for each mix.

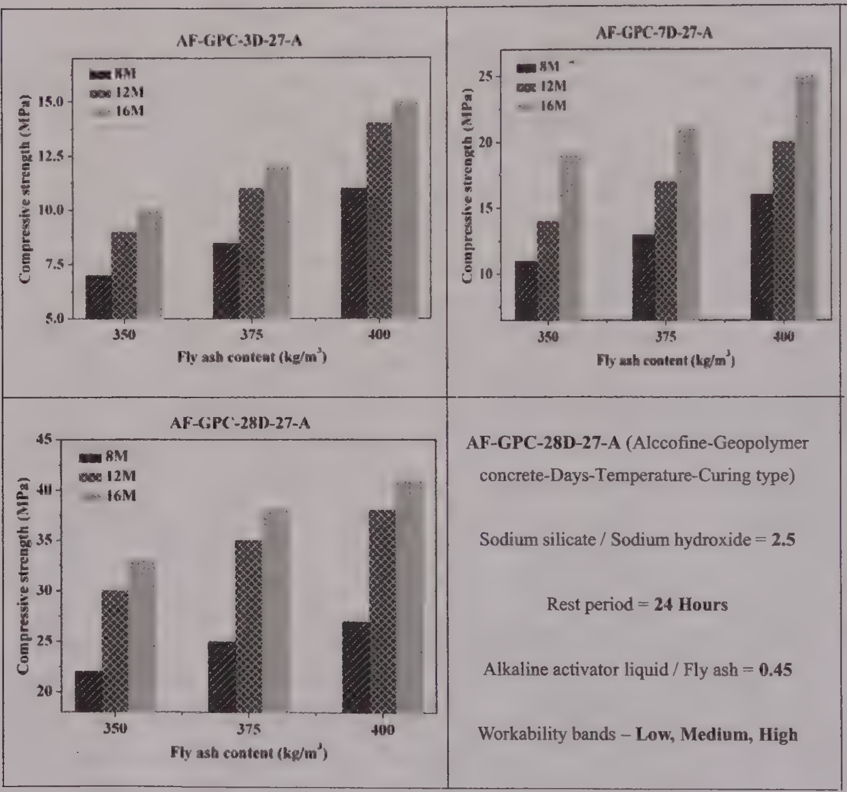
Table 4. 4: Compressive strength of FA based GPC

Mixture	Average compressive strength (MPa)		
	3 days	7 days	28 days
M1FAGC	7.15	12.15	22.09
M2FAGC	8.05	14.17	25.12
M3FAGC	9.10	15.20	27.15
M4FAGC	8.04	14.06	30.05
M5FAGC	10.19	17.11	35.10

M6FAGC	12.10	20.10	38.17
M7FAGC	10.11	19.06	33.12
M8FAGC	12.05	21.05	38.18
M9FAGC	15.09	25.10	41.00
M10FAGC	14.18	23.63	26.25
M11FAGC	15.80	26.33	29.25
M12FAGC	17.82	29.70	33.02
M13FAGC	19.85	33.08	36.75
M14FAGC	21.06	35.10	39.00
M15FAGC	22.28	37.13	41.25
M16FAGC	26.33	43.88	48.75
M17FAGC	27.95	46.58	51.75
M18FAGC	29.57	49.28	54.75
M19FAGC	19.22	32.15	35.20
M20FAGC	21.65	36.35	39.15
M21FAGC	23.43	39.17	44.08
M22FAGC	28.26	47.05	49.10
M23FAGC	29.43	49.12	52.05
M24FAGC	31.20	52.06	55.11
M25FAGC	37.22	62.17	65.12
M26FAGC	39.05	65.10	69.11
M27FAGC	40.80	68.08	73.00

Figs. 4.8 to 4.10 show the variation in compressive strength with fly ash content for geopolymer concrete specimens prepared at different temperatures and molarities.

The mixes from M1FAGC to M27FAGC were used to develop the AF-GPC graphs which were further used to develop the process of mix designs for geopolymer concrete. Table 4.4 reports the 3, 7 and 28 days compressive strength with varied temperature and binder content.



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

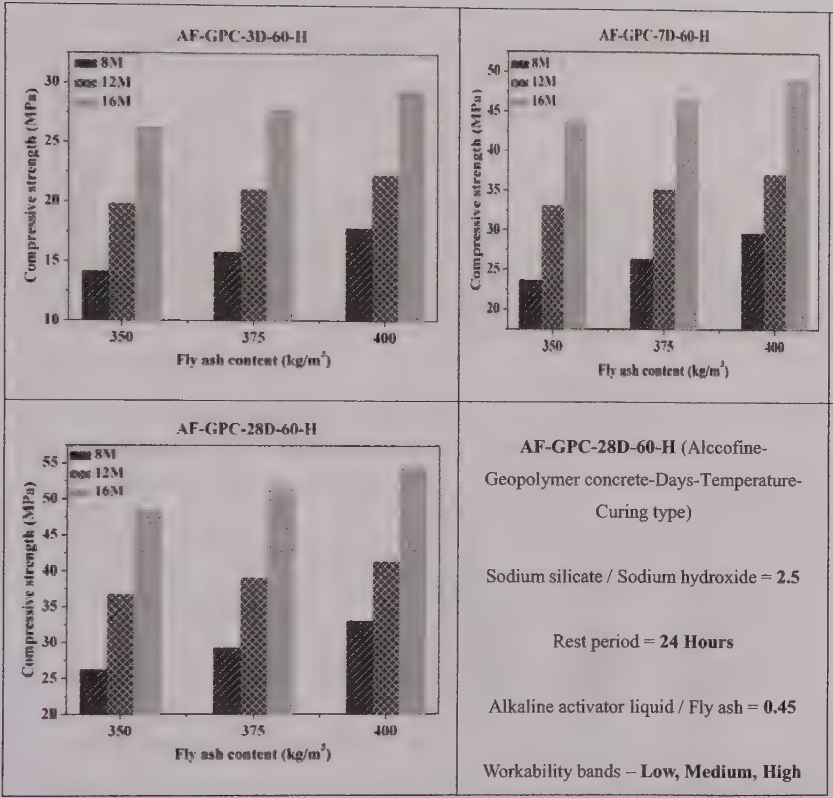
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.8: Compressive strength with varying fly ash content and curing period at 27^{O}C curing temperature



AF-GPC-28D-60-H (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

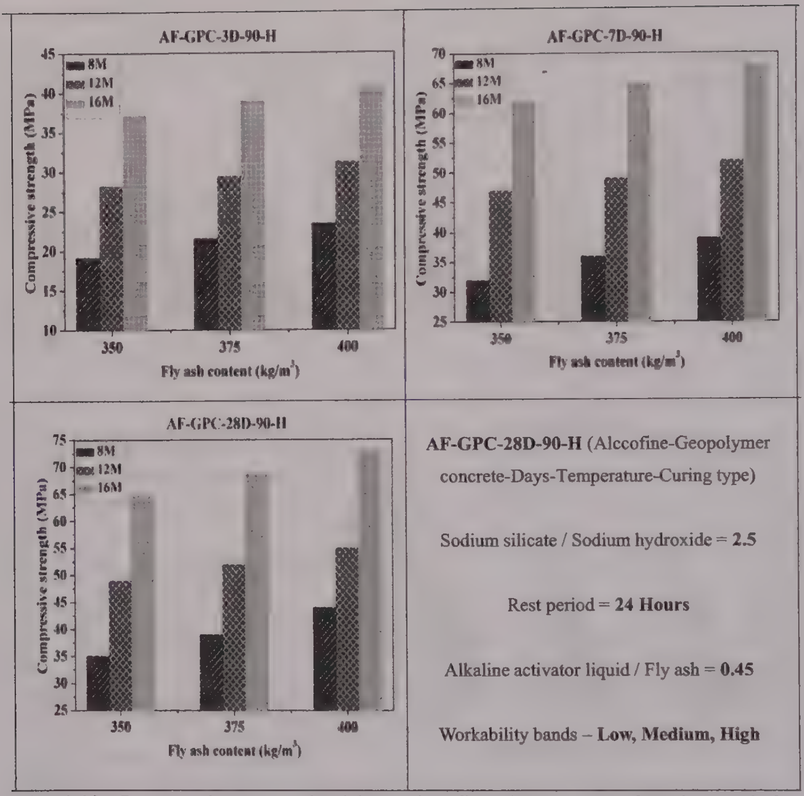
Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.9: Compressive strength with varying fly ash content and curing period at $60^{0}C$ curing temperature

It can be observed from Figs. 4.8 to 10 that the compressive strength of the geopolymer concrete increased with the increase in temperature, NaOH molarity and fly ash content. Comparing the 28 days strength of the Mixes M9FAGC, M18FAGC and M27FAGC which were cured at 27° C, 60° C and 90° C, a maximum of 41 MPa, 54.75 MPa and 73 MPa compressive strengths were obtained.



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.10: Compressive strength with varying fly ash content and curing period at 90°C curing temperature

The shape of the strength development curves in Figs. 4.8 to 4.10 indicates that with the increase in NaOH molarity, curing temperature and fly ash content, a remarkable higher strength can be achieved. For example, the increase in strength was about 32-34% when temperature changed from 27° C to 60° C (M9FAGC & M18FAGC) and about 77-79% when the changed was 27° C to 90° C (M9FAGC & M27FAGC) at the curing age of 28 days.

Also, when fly ash content changed from 350 kg/m³ to 375 kg/m³ and 400 kg/m³, increased in strengths were 15-17% and 7-9%, 7 days compressive strength of the mixes M1FAGC to M3FAGC which were cured at 27° C and 8M NaOH solution. It was further influenced by temperature as when 7 days strength of the heat cured specimens with same fly ash content were compared this effect increased from 12-14% and 20-23%, respectively, for reference mixes M19FAGC to M21FAGC were used. This shows that fly ash content and temperature plays an important role into the matrix.

When molarity changed from 8M to 12M and 16M compressive strength increased from 39 MPa to 52MPa and 69MPa, mixes M20FAGC, M23FAGC and M26FAGC were compared. The rate of increase in strengths were 33-34% and 76-78%, respectively. This change was significantly more when compared with ambiently cured specimens. Also, combined effect of molarity and temperature was similar to the effects of fly ash contents and temperatures as discussed above.

Further, it is worth noting that all the mixes show a still developing strength curves and promise to provide even higher strength above 400 Kgs of fly ash. This phenomenon is true for all the parameters considered in this study for GPC. It can also be seen from the Table 4.4 that the rate of the strength development after 7 days is less, unlike conventional concrete. Normally, the rate of gain of strength with ages was 68 – 72 % and 10-13% from 3 days to 7 days and 7 days to 28 days, respectively, for the specimens which were heat cured. However, for ambient cured GPC specimens this change was different, 66 – 71 % and 62-66% from 3 days to 7 days and 7 days to 28 days, respectively.

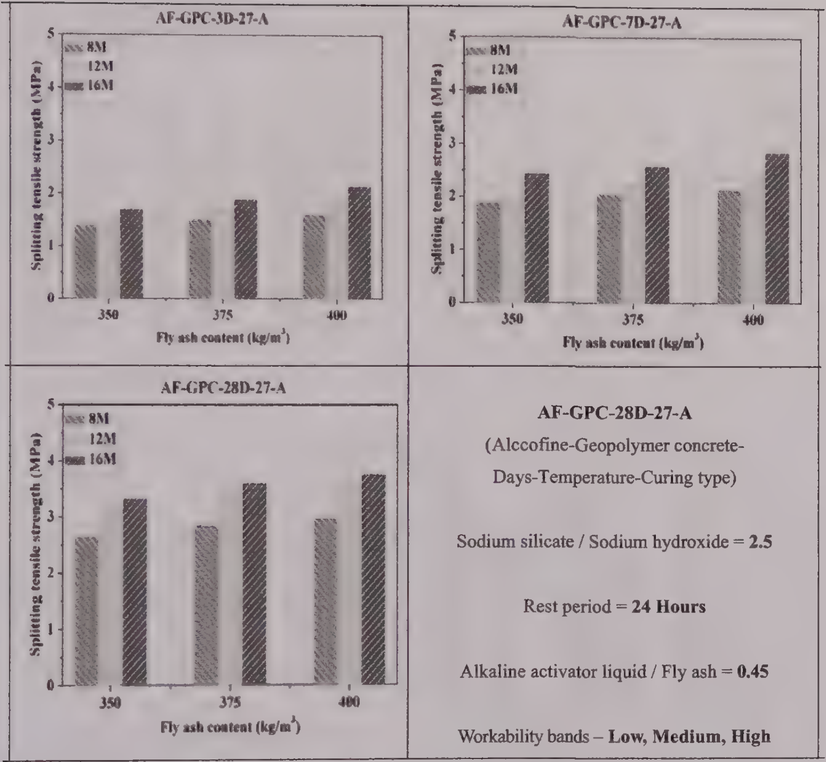
4.3.1.2 Split tensile strength

As split tensile strength of the GPC was influenced by NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine therefore, it was studied and measured at the ages of 3, 7 and 28 days. The average split tensile strength of five specimens is reported in the Table 4.5 for each mix and showed with the help of Figs. 4.11 to 4.13.

Table 4. 5: Split tensile strength of FA based GPC

Mixture	Average split tensile strength (MPa)		
	3 days	7 days	28 days
M1FAGC	1.39	1.88	2.64
M2FAGC	1.50	2.05	2.84
M3FAGC	1.60	2.13	2.96
M4FAGC	1.50	2.05	3.14
M5FAGC	1.70	2.29	3.43

M6FAGC	1.88	2.50	3.59
M7FAGC	1.70	2.43	3.32
M8FAGC	1.88	2.57	3.59
M9FAGC	2.13	2.84	3.75
M10FAGC	2.06	2.75	2.92
M11FAGC	2.19	2.92	3.10
M12FAGC	2.35	3.13	3.32
M13FAGC	2.49	3.32	3.52
M14FAGC	2.58	3.43	3.64
M15FAGC	2.66	3.54	3.76
M16FAGC	2.92	3.89	4.13
M17FAGC	3.02	4.03	4.27
M18FAGC	3.12	4.16	4.41
M19FAGC	2.45	3.26	3.43
M20FAGC	2.61	3.48	3.64
M21FAGC	2.73	3.64	3.90
M22FAGC	3.04	4.05	4.14
M23FAGC	3.11	4.14	4.28
M24FAGC	3.21	4.28	4.42
M25FAGC	3.55	4.73	4.86
M26FAGC	3.64	4.86	5.02
M27FAGC	3.74	4.98	5.18



AF-GPC-28D-27-A

(Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

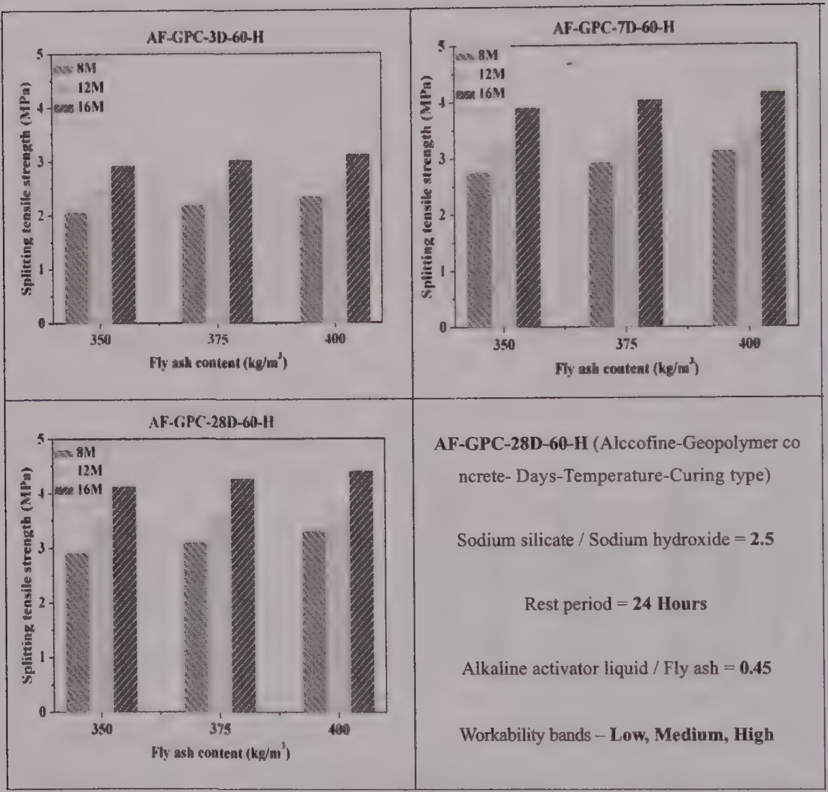
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.11: Split tensile strength with varying fly ash content and curing period at 27^{O}C curing temperature



AF-GPC-28D-60-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

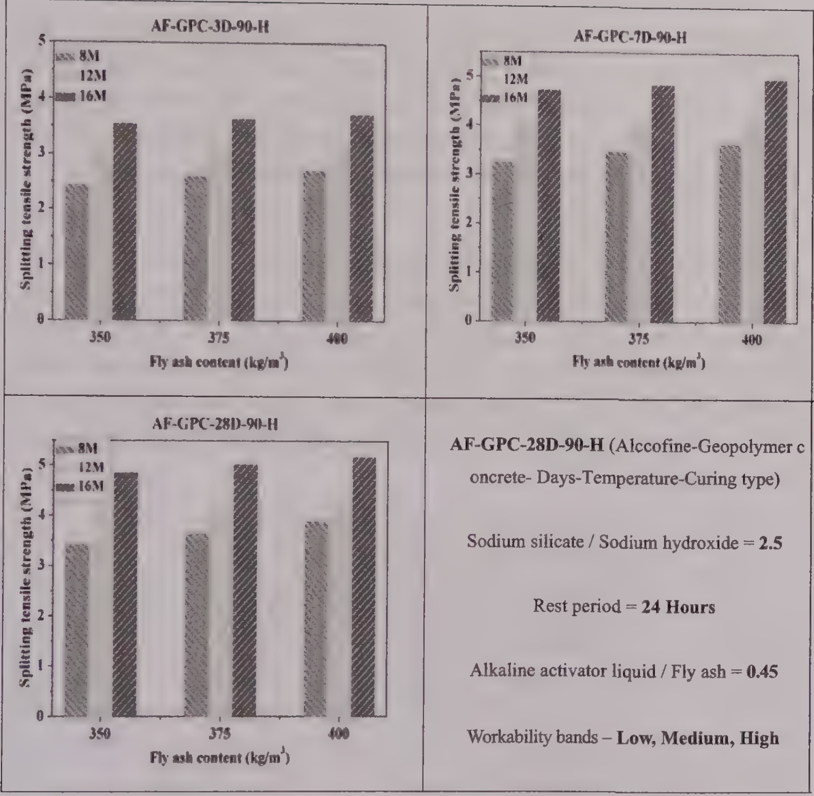
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.12: Split tensile strength with varying fly ash content and curing period at 60°C curing temperature



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.13: Split tensile strength with varying fly ash content and curing period at 90^{O}C curing temperature

It was observed that the split tensile strength increased with the increase in NaOH molarities, fly ash contents and temperatures in the system. For example, the increase in strength was about 13-15% when molarity changed from 8M to 12M (M21FAGC & M24FAGC) and about 32-33% when changed from 8M to 16M (M21FAGC & M27FAGC) for the same fly ash content at the age of 28 days. Further, the change noticed due to change in fly ash content was about 3–5% and 6-7% when mix M16FAGC was compared with M17FAGC and M18FAGC. Temperature had significant effect on the early and final age strengths of the concrete as it helps in the polymerisation process. It was observed that with the increase in the temperature the split tensile strength increased significantly in all the cases. Also, for all the specimens, cured ambiently, the increase in strength was noticed with the age. Further, this change was more significant at early age when heat curing was adopted. This might be due to geopolymer mechanism which involved polymerisations that initiated with the high temperature. In general, the effects on the split tensile strength with different variables were very much similar to the effects of compressive strength as has been discussed above.

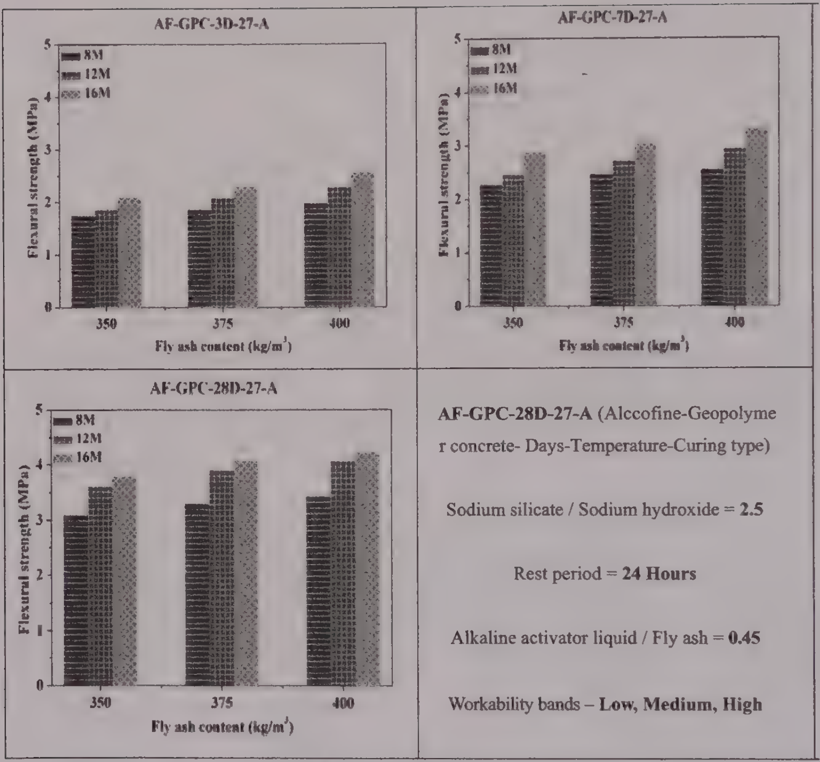
4.3.1.3 Flexural strength

The flexural strength of the geopolymer specimens was studied in terms of modulus of rupture and the influences of NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine into GPC on the flexural strength were carried out. Similar to other tests, the flexural strength was measured after 3, 7 and 28 days. The specimens were cured at 27° C, 60° C and 90° C. The average flexural strength in the Table 4.6 is average over three specimens. Figs. 4.14 to 4.16 shows the flexural strength for geopolymer concrete identical specimens.

Table 4. 6: Flexural strength of FA based GPC

Mixture	Average flexural strength (MPa)		
	3 days	7 days	28 days
M1FAGC	1.75	2.29	3.10
M2FAGC	1.87	2.47	3.30
M3FAGC	1.98	2.56	3.43
M4FAGC	1.87	2.47	3.61
M5FAGC	2.09	2.72	3.90
M6FAGC	2.29	2.95	4.07
M7FAGC	2.09	2.88	3.79
M8FAGC	2.29	3.02	4.07
M9FAGC	2.56	3.30	4.23
M10FAGC	2.48	3.21	3.38
M11FAGC	2.62	3.39	3.57
M12FAGC	2.79	3.60	3.79
M13FAGC	2.94	3.80	4.00
M14FAGC	3.03	3.91	4.12
M15FAGC	3.11	4.02	4.24

M16FAGC	3.39	4.37	4.61
M17FAGC	3.49	4.50	4.75
M18FAGC	3.59	4.63	4.88
M19FAGC	2.89	3.73	3.90
M20FAGC	3.07	3.96	4.12
M21FAGC	3.19	4.12	4.38
M22FAGC	3.50	4.52	4.62
M23FAGC	3.58	4.62	4.76
M24FAGC	3.69	4.76	4.89
M25FAGC	4.03	5.20	5.32
M26FAGC	4.12	5.32	5.48
M27FAGC	4.22	5.44	5.64



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

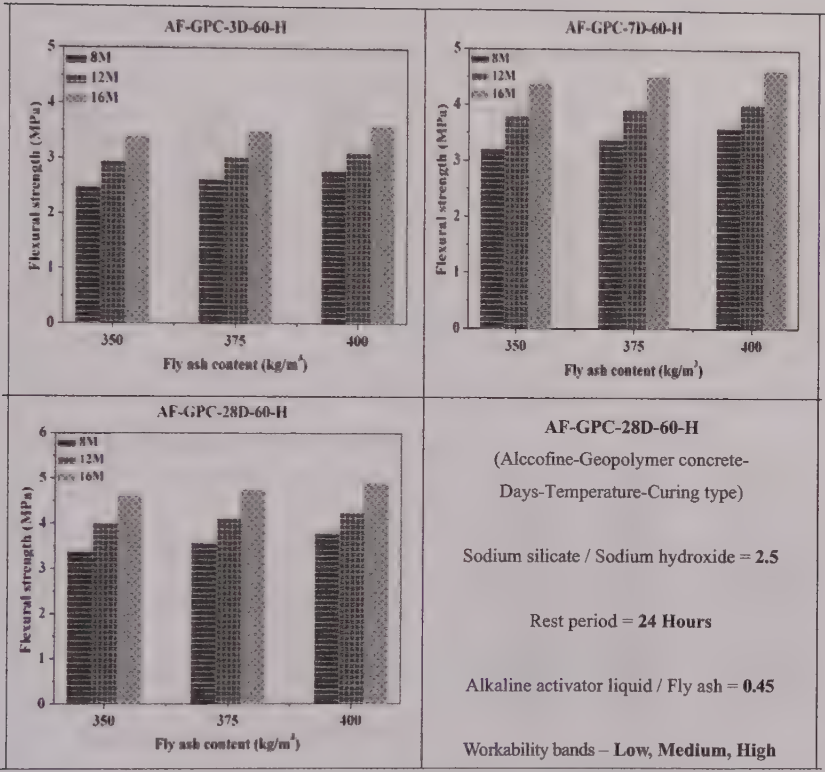
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.14: Flexural strength with varying fly ash content and curing period at 27^{O}C curing temperature



AF-GPC-28D-60-H

(Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

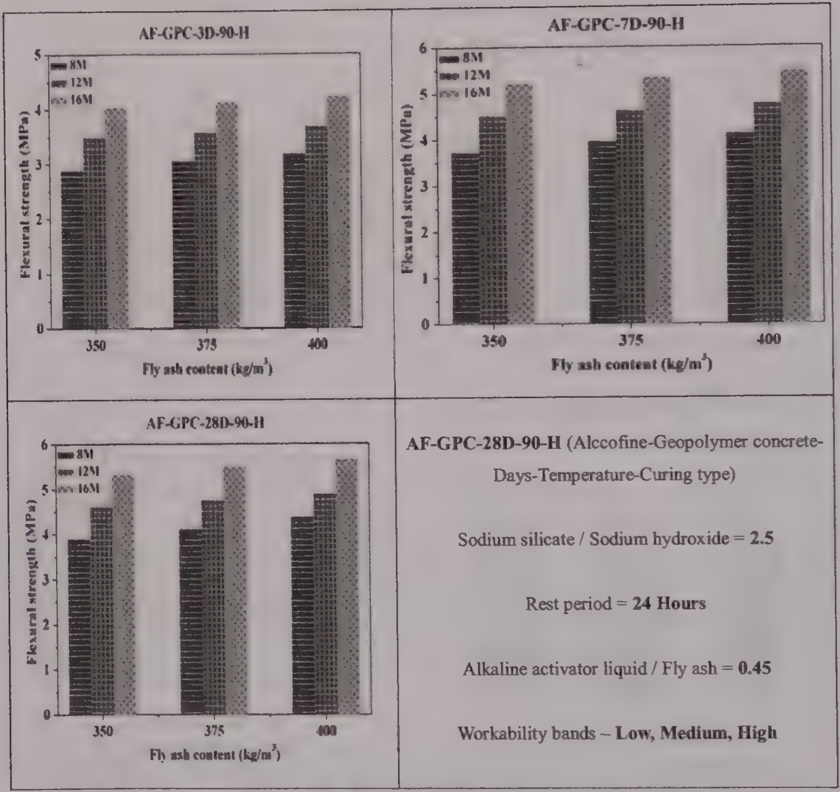
Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.15: Flexural strength with varying fly ash content and curing period at 60°C curing temperature

Flexural strength of the geopolymer concrete specimens had similar effects as has been discussed in the case of compressive and split tensile strength. For example, at 7 days, the strength of mixes M21FAGC, M24FAGC, M27FAGC, M16FAGC, M17FAGC and M18FAGC were 4.12, 4.76, 5.44, 4.37, 4.50 and 4.63 MPa, respectively. Although the strength increment was on higher side when fly ash content, molarity and temperature increased, yet the best results in terms of maximum strength was obtained by the specimens with 400kg fly ash, 16M and at 90^0 curing temperature.



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.16: Flexural strength with varying fly ash content and curing period at 90°C curing temperature

The flexural strength increased with the increase in fly ash content at different ages and the trend was similar to the trend of conventional concrete. The only difference being the percentage increase of strength was found to be 5-6% and 10-11% for ambiently cured and 5-6% and 12-13% for heat cured specimens, respectively, when fly ash content increased from 350 kg/m^3 to 375 kg/m^3 and 400 kg/m^3 at the age of 28 days. Similarly, for all the specimens in the system 3 and 7 days flexural strength of the geopolymer concrete specimens were 74-75% and 96-97% of its 28 days flexural strength. Also, when the temperature increased from the 27°C to 60°C and 60°C to 90°C, the strength increased by 40-41% and 17-18%, respectively, at all the ages.

4.3.2 Rice Husk Ash Based Geopolymer Concrete

The effects of different parameters on the structural properties of the rice husk ash based geopolymer concrete has been discussed below.

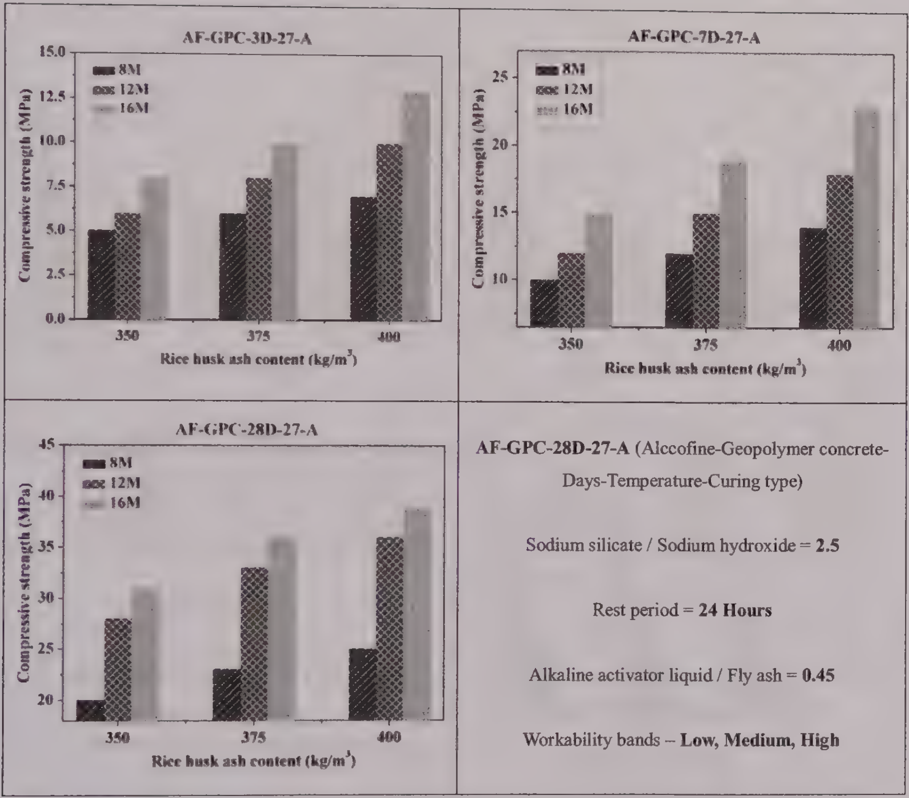
4.3.2.1 Compressive strength

Although, results of the RHA based geopolymer concrete was somewhat in the same range as observed for FA based GPC yet the influences of NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine into RHA based geopolymer concrete on the compressive strength were studied. The specimens were cured at 27° C, 60° C and 90° C and compressive strength was measured after 3, 7 and 28 days. The average compressive strength of five specimens in Table 4.7 is average over five specimens. Figs. 4.17 to 4.19 shows the compressive strength for RHA based geopolymer concrete specimens at 27° C, 60° C and 90° C.

Table 4. 7: Compressive strength of RHA based GPC

Mixture	Average compressive strength (MPa)		
	3 days	7 days	28 days
M1RHAGC	5.02	10.15	20.01
M2RHAGC	6.10	12.23	23.08
M3RHAGC	7.11	15.04	25.22
M4RHAGC	6.09	12.17	28.34
M5RHAGC	8.20	15.35	33.29
M6RHAGC	10.07	19.20	36.17
M7RHAGC	8.25	14.33	31.60
M8RHAGC	10.23	18.15	36.05
M9RHAGC	13.02	23.06	39.00
M10RHAGC	12.18	21.63	24.25
M11RHAGC	13.80	24.33	27.25
M12RHAGC	15.11	27.76	31.00
M13RHAGC	17.85	31.08	34.75
M14RHAGC	19.06	33.10	37.00
M15RHAGC	20.28	35.13	39.25
M16RHAGC	24.33	41.88	46.75

M17RHAGC	25.95	44.58	49.75
M18RHAGC	27.57	47.28	52.75
M19RHAGC	17.20	30.05	33.12
M20RHAGC	19.62	34.18	37.10
M21RHAGC	21.41	37.02	42.11
M22RHAGC	26.22	45.14	47.15
M23RHAGC	27.44	47.19	50.16
M24RHAGC	29.27	50.08	53.12
M25RHAGC	35.23	60.20	63.04
M26RHAGC	37.08	63.19	67.03
M27RHAGC	38.83	66.13	71.00



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

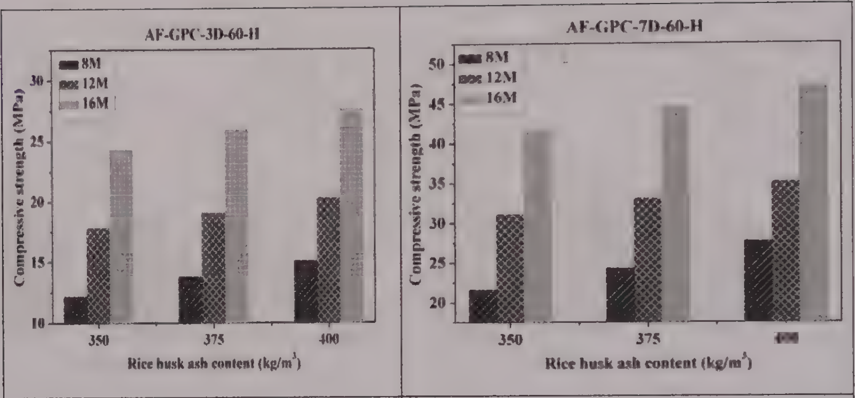
Rest period = **24 Hours**

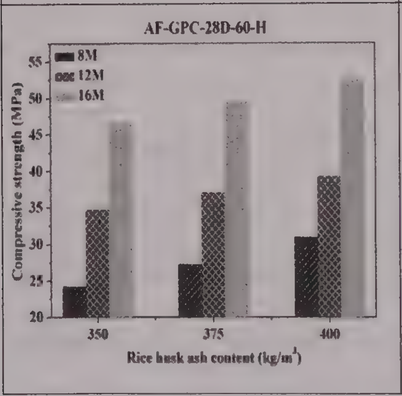
Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.17: Compressive strength with varying rice husk ash content and curing period at 27^{0}C curing temperature

Fig. 4.17 shows the effects of various parameters on the geopolymer concrete prepared using alccofine and rice husk ash at ambient temperature.





AF-GPC-28D-60-H (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

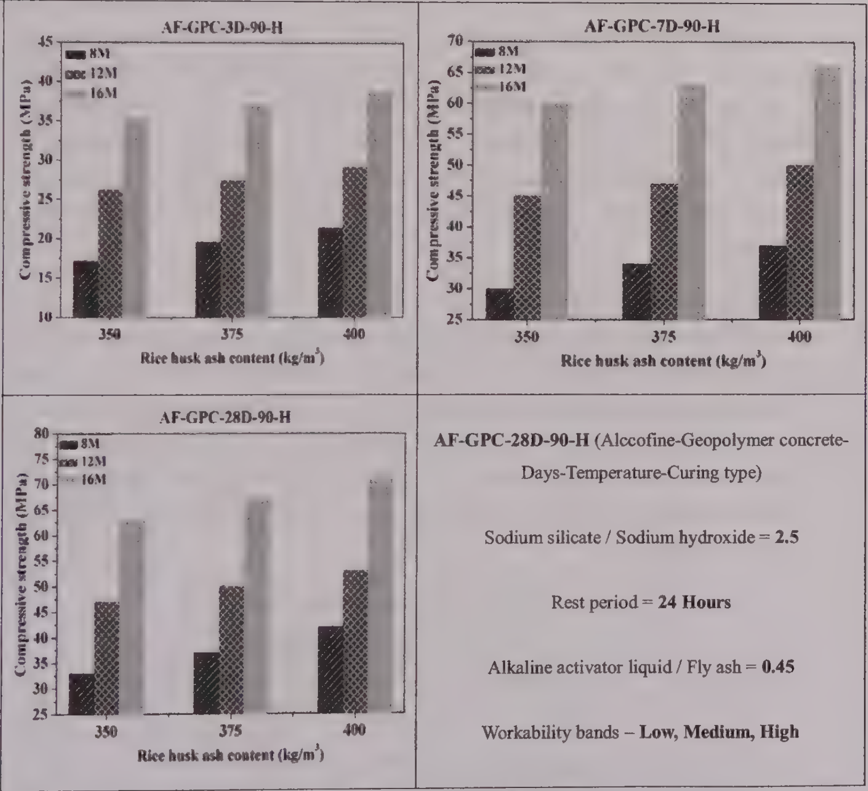
Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.18: Compressive strength with varying rice husk ash content and curing period at 60^{O}C curing temperature

It can be concluded from the Fig. 4.17 that the compressive strength increased with the increase in rice husk content, sodium hydroxide molarity, temperature and age. For example, at 28 days, the strength of the mixes M7RHAGC, M8RHAGC and M9RHAGC were 31.0, 36.0 and 39.0 MPa, respectively. The increase in strength for the above mixes were 19-20% and 8-9% when RHA content changed from 350 to 375 and 375 to 400 Kg/m^3 into the matrix. The strength was lower when the mixes with same RHA content and molarity were compared at ambient and elevated heat temperature in the similar way as discussed in FA based GPC.

Further, the increase in strength recorded was 40% and 10% when the NaOH molarity changed from the 8M to 12M and 12M to 16M (M1RHAGC, M4RHAGC and M7RHAGC were compared for reference). Also, when 3, 7 and 28 days strength of the mix M9RHAGC was compared, it was observed that the increase in the strength were 80% and 200%. Further, 3 days and 7 days compressive strength recorded were 34% and 60% of the total ultimate strength achieved.



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.19: Compressive strength with varying rice husk ash content and curing period at 90^{0}C curing temperature

Figs. 4.17 to 4.19 show the slope between compressive strength and rice husk ash content for the mixes tabulated in Table 4.7. Figs. 4.18 and 4.19 show the relationship for specimens cured at 60°C and 90°C, respectively. It was observed that compressive strength increased with increase in temperature and the increment had same effects with the increase in molarity, age of curing and RHA content. The only difference was of being the percentage increase in compressive strength i.e. 3 days compressive strength of heat cured geopolymer concrete specimens was found to be 51-53% and 7 days compressive strength was found to be 92-93% of its 28 days strength whereas for conventional concrete the normal 7 days strength can be considered as around 65-70% of the 28 days strength. Also, similar behaviour was observed for fly ash based geopolymer concrete as already been discussed. Further, the curing temperature had significant effect on the compressive strength. For example, when 7 days strength of M1RHAGC, M10RHAGC and M19RHAGC were compared, a percentage increase of 117% and 39% was observed. This meant that high strength can be achieved only after 7 days if heat curing adopted. Also, higher concentration of the alkaline solution increased the strength and the same trend was observed for fly ash based geopolymer concrete.

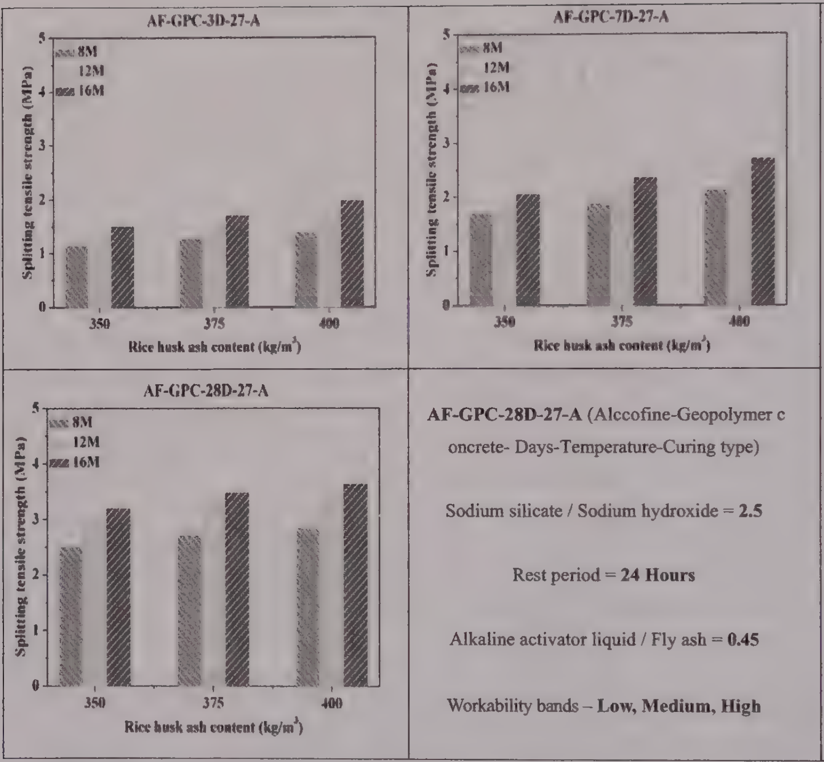
4.3.2.2 Split tensile strength

As discussed above that split tensile strength is the important structural property therefore, the influences of NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine into the geopolymer concrete on the split tensile strength were studied. The split tensile strength was measured after 3, 7 and 28 days. Table 4.8 shows the average split tensile strength of the five specimens for all the twenty-seven mixes. Figs. 4.20 to 4.22 shows the split tensile strength for geopolymer concrete specimens cured at 27° C, 60° C and 90° C.

Table 4. 8: Split tensile strength of RHA based GPC

Mixture	Average split tensile strength (MPa)		
	3 days	7 days	28 days
M1RHAGC	1.15	1.70	2.50
M2RHAGC	1.27	1.88	2.71
M3RHAGC	1.39	2.13	2.84
M4RHAGC	1.27	1.88	3.03
M5RHAGC	1.50	2.13	3.32
M6RHAGC	1.70	2.43	3.48

M7RHAGC	1.50	2.05	3.20
M8RHAGC	1.70	2.36	3.48
M9RHAGC	1.97	2.71	3.64
M10RHAGC	1.89	2.62	2.79
M11RHAGC	2.03	2.80	2.98
M12RHAGC	2.14	3.01	3.20
M13RHAGC	2.35	3.21	3.42
M14RHAGC	2.44	3.32	3.54
M15RHAGC	2.52	3.44	3.66
M16RHAGC	2.80	3.79	4.04
M17RHAGC	2.90	3.93	4.18
M18RHAGC	3.00	4.06	4.32
M19RHAGC	2.30	3.14	3.32
M20RHAGC	2.48	3.37	3.54
M21RHAGC	2.60	3.54	3.80
M22RHAGC	2.91	3.95	4.05
M23RHAGC	2.99	4.05	4.19
M24RHAGC	3.10	4.19	4.33
M25RHAGC	3.44	4.64	4.77
M26RHAGC	3.54	4.77	4.94
M27RHAGC	3.63	4.90	5.10



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

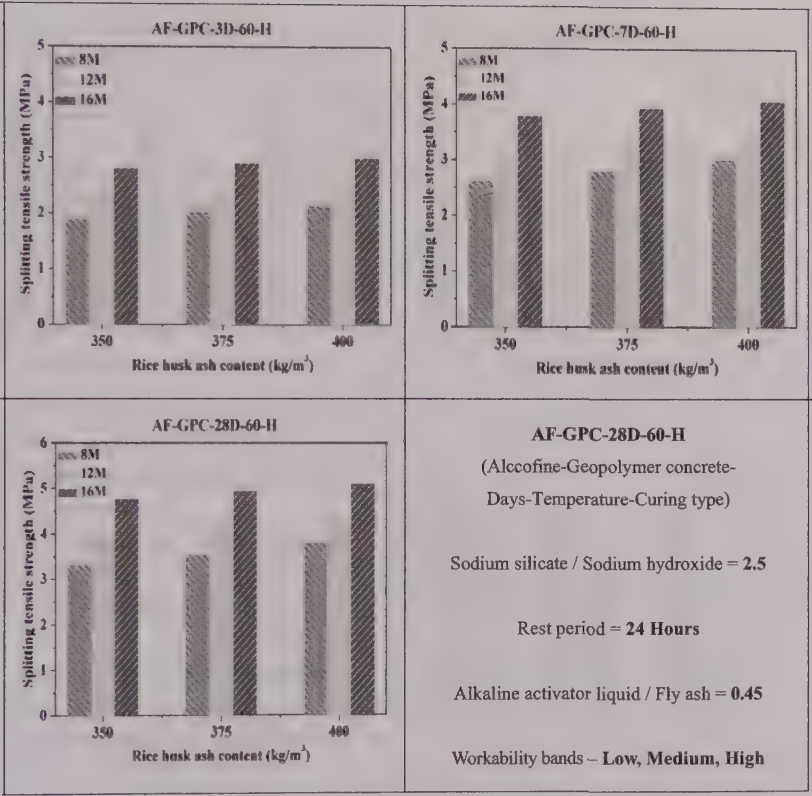
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.20: Split tensile strength with varying rice husk ash content and curing period at 27^{0}C curing temperature



AF-GPC-28D-60-H

(Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

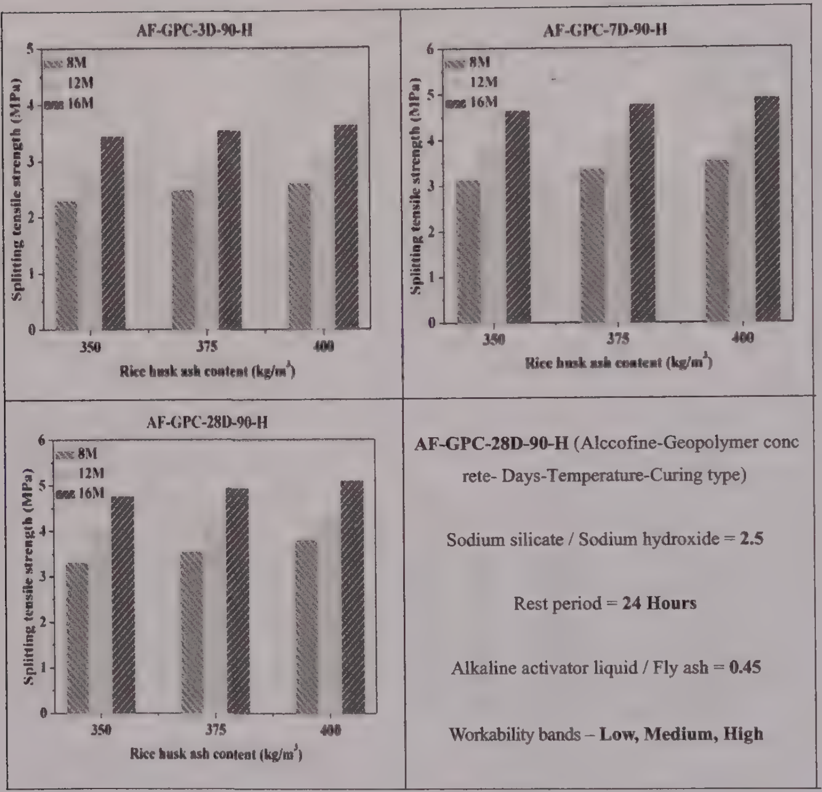
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.21: Split tensile strength with varying rice husk ash content and curing period at $60^{0}C$ curing temperature



AF-GPC-28D-90-H (Alccofine-Geopolymer conc rete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.22: Split tensile strength with varying rice husk ash content and curing period at 90^{0}C curing temperature

Split tensile strength of the geopolymer specimens had similar trends as was observed in the compressive strength of the geopolymer concrete activated from alccofine and alkaline solution. For example, at 7 days, the strength of mixes M21RHAGC, M24RHAGC, M27RHAGC, M16RHAGC, M17RHAGC and M18RHAGC were 3.54, 4.19, 4.90, 3.79, 3.93 and 4.06 MPa, respectively. Split tensile strength increased when RHA content, NaOH molarity and curing temperature increased, however, ultimate strength was achieved with 400kg rice husk ash, 16M and at heat curing. The trend of increasing the strength with rice husk ash, age content was observed just like conventional concrete except he only difference being the percentage increase of strength was found to be 7% and 14% when rice husk ash content increased from 350 kg/m^3 to 375 kg/m^3 and 400 kg/m^3. Similarly, the 3 and 7 days strength of the geopolymer specimens were 65-72% and 92-95% of its 28 days strength for all the specimens in the system when heat curing adopted. Further, this change was 44-46% and 63-66% for ambient cured specimens. Also, when the temperature increases from the 27°C to 60°C and 60°C to 90°C, the 7 days strength increased rapidly and a change of 53-55% and 19-20% was observed.

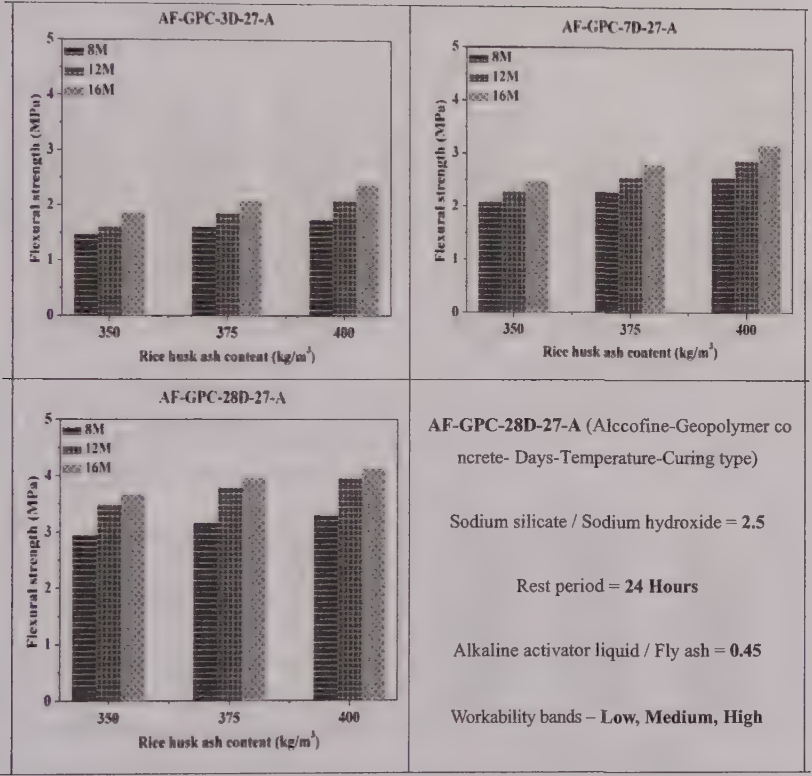
4.3.2.3 Flexural strength

Flexural strength has equal importance like other structural properties therefore, it is important to study the influences of NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine into geopolymer concrete on the flexural strength. The average flexural strength in the Table 4.9 is average over three specimens which were cured at 27° C, 60° C and 90° C. Figs. 4.23 to 4.25 shows the flexural strength for geopolymer concrete specimens at the age of 3, 7 and 28 days.

Table 4. 9: Flexural strength of RHA based GPC

Mixture	**Average flexural strength (MPa)**		
	3 days	**7 days**	**28 days**
M1RHAGC	1.48	2.09	2.95
M2RHAGC	1.62	2.29	3.17
M3RHAGC	1.75	2.56	3.30
M4RHAGC	1.62	2.29	3.49
M5RHAGC	1.87	2.56	3.79
M6RHAGC	2.09	2.88	3.96
M7RHAGC	1.87	2.47	3.67
M8RHAGC	2.09	2.80	3.96
M9RHAGC	2.38	3.17	4.12
M10RHAGC	2.30	3.07	3.25
M11RHAGC	2.45	3.26	3.45
M12RHAGC	2.56	3.47	3.67
M13RHAGC	2.79	3.68	3.89
M14RHAGC	2.88	3.80	4.01
M15RHAGC	2.97	3.91	4.13

M16RHAGC	3.26	4.27	4.51
M17RHAGC	3.36	4.41	4.66
M18RHAGC	3.47	4.54	4.79
M19RHAGC	2.74	3.61	3.79
M20RHAGC	2.92	3.85	4.01
M21RHAGC	3.05	4.01	4.28
M22RHAGC	3.38	4.43	4.52
M23RHAGC	3.45	4.52	4.67
M24RHAGC	3.57	4.67	4.80
M25RHAGC	3.92	5.11	5.24
M26RHAGC	4.01	5.24	5.40
M27RHAGC	4.11	5.36	5.56



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

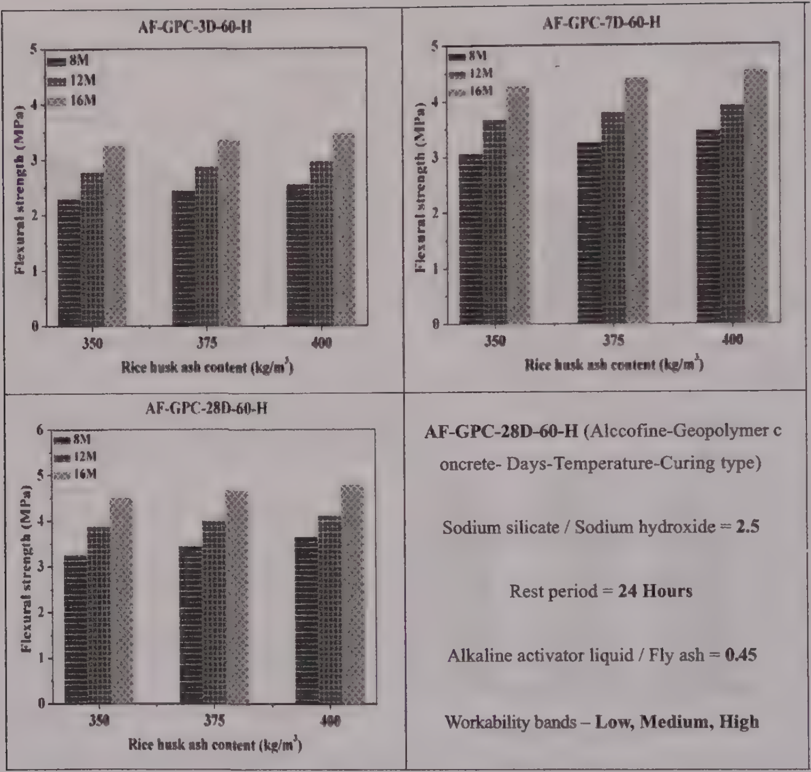
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.23: Flexural strength with varying rice husk ash content and curing period at 27^{O}C curing temperature



AF-GPC-28D-60-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

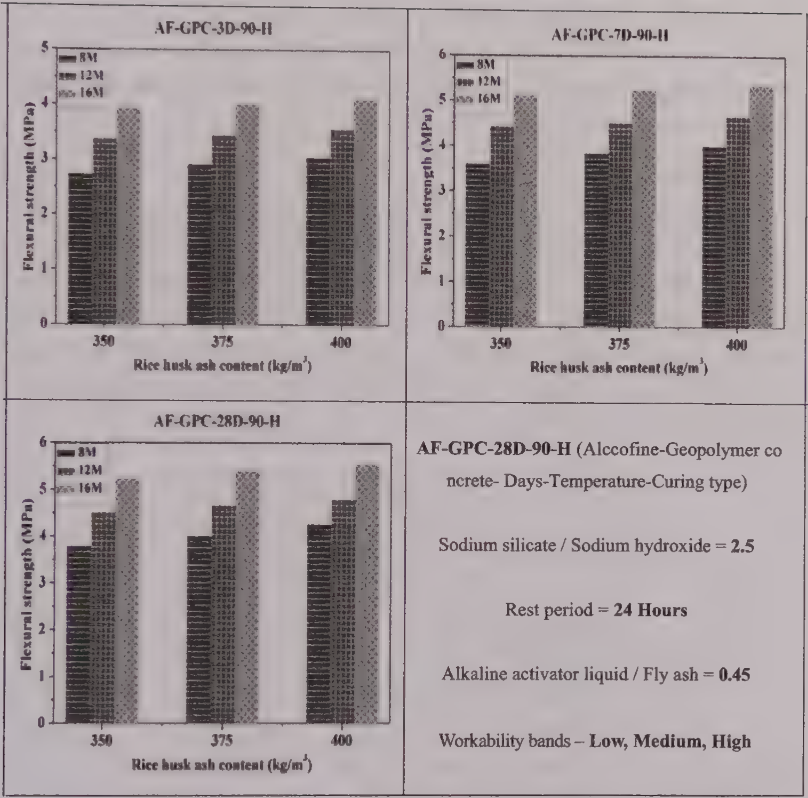
Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.24: Flexural strength with varying rice husk ash content and curing period at 60°C curing temperature

It was observed that with the increase in NaOH molarity, RHA content and temperature in the system, the flexural tensile strength increased. For example, the increase in strength was about 11-13% when molarity changes from 8M to 12M (M21RHAGC & M24RHAGC) and about 29-31% when 8M to 16M (M21RHAGC & M27RHAGC) after 28 days. Further the change noticed due to change in RHA content after 28days, was about 3–4% and 5-6% when mix M16RHAGC is compared with M17RHAGC and M18RHAGC.



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.25: Flexural strength with varying rice husk ash content and curing period at 90^{o}C curing temperature

Temperature has significant effect on the early and final age strength of the concrete as it helps in the polymerisation and hydration process. It was observed that with the increase in the curing temperature the flexural strength increased significantly. For example, when 7 days strength of M2RHAGC compared with M11RHAGC and M20RHAGC, a change of 42-43% and 67-69% was observed in flexural strength. The trend of increasing the flexural strength at different ages was similar to the trend of split tensile strength. Further, this change of increased in strength was more significant at early age when heat curing was adopted.

4.3.3 GGBS Based Geopolymer Concrete

The effects of different parameters on the structural properties such as compressive, split tensile and flexural strengths of the ground granulated blast furnace slag based geopolymer concrete have been discussed below.

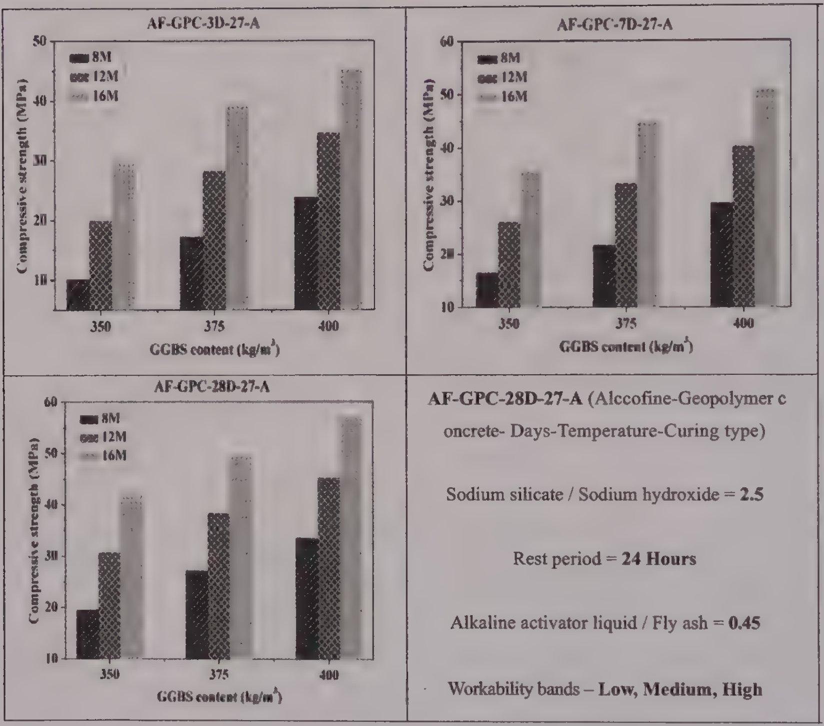
4.3.3.1 Compressive strength

It is the most common and indicator of structural property of the concrete which is also correlated with the other properties. Similar to FA and RHA based GPC, the influences of NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine into GGBS based geopolymer concrete on the compressive strength were studied. Table 4.10 shows the average compressive strength over five identical specimens for each mix. Figs. 4.26 to 4.28 show the compressive strength for geopolymer concrete specimens cured at the ages of 27° C, 60° C and 90° C and at the ages of 3, 7 and 28 days.

Table 4. 10: Compressive strength of GGBS based GPC

Mixture	**Average compressive strength (MPa)**		
	3 days	**7 days**	**28 days**
M1GGBSGC	10.10	16.55	19.50
M2GGBSGC	17.25	21.65	27.20
M3GGBSGC	23.85	29.45	33.50
M4GGBSGC	19.88	26.05	30.67
M5GGBSGC	28.23	33.20	38.31
M6GGBSGC	34.55	40.15	45.25
M7GGBSGC	29.65	35.55	41.84
M8GGBSGC	39.20	44.75	49.42
M9GGBSGC	45.25	50.85	57.00
M10GGBSGC	31.55	38.00	40.95
M11GGBSGC	38.70	43.10	48.65
M12GGBSGC	45.30	50.90	54.95
M13GGBSGC	41.73	47.90	52.52
M14GGBSGC	50.08	55.05	60.16
M15GGBSGC	56.40	62.00	67.10
M16GGBSGC	51.43	57.33	63.62

M17GGBSGC	60.98	66.53	71.20
M18GGBSGC	67.03	72.63	78.78
M19GGBSGC	35.80	42.25	45.20
M20GGBSGC	42.95	47.35	52.90
M21GGBSGC	49.55	55.15	59.20
M22GGBSGC	45.58	51.75	56.37
M23GGBSGC	53.93	58.90	64.01
M24GGBSGC	60.25	65.85	70.95
M25GGBSGC	55.35	61.25	67.54
M26GGBSGC	64.90	70.45	75.12
M27GGBSGC	70.95	76.55	82.70



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

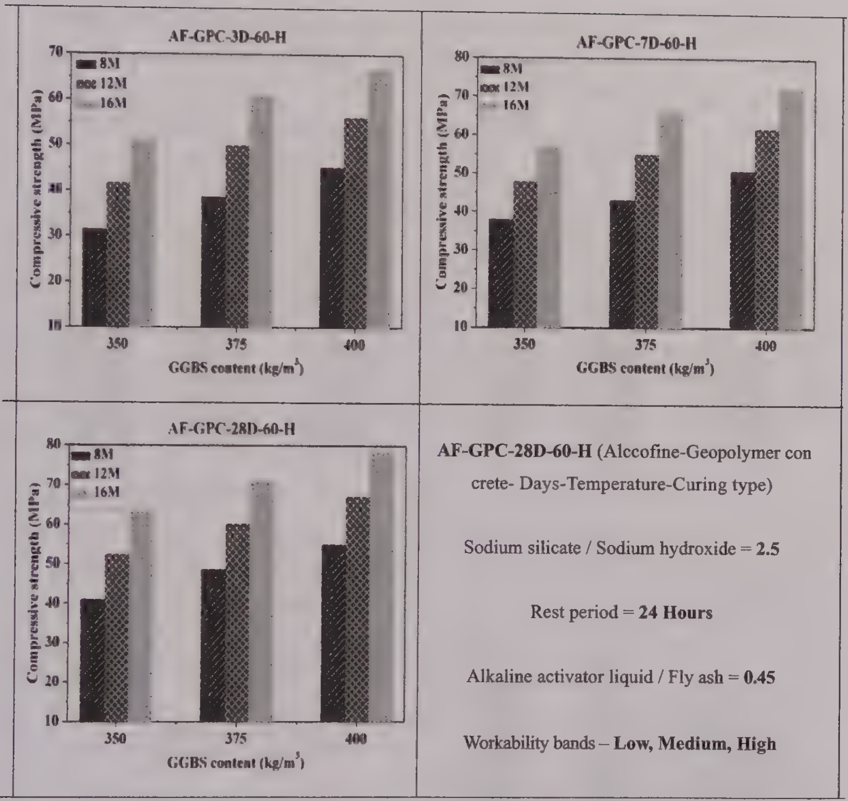
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.26: Compressive strength with varying GGBS content and curing period at 27^{O}C curing temperature



AF-GPC-28D-60-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

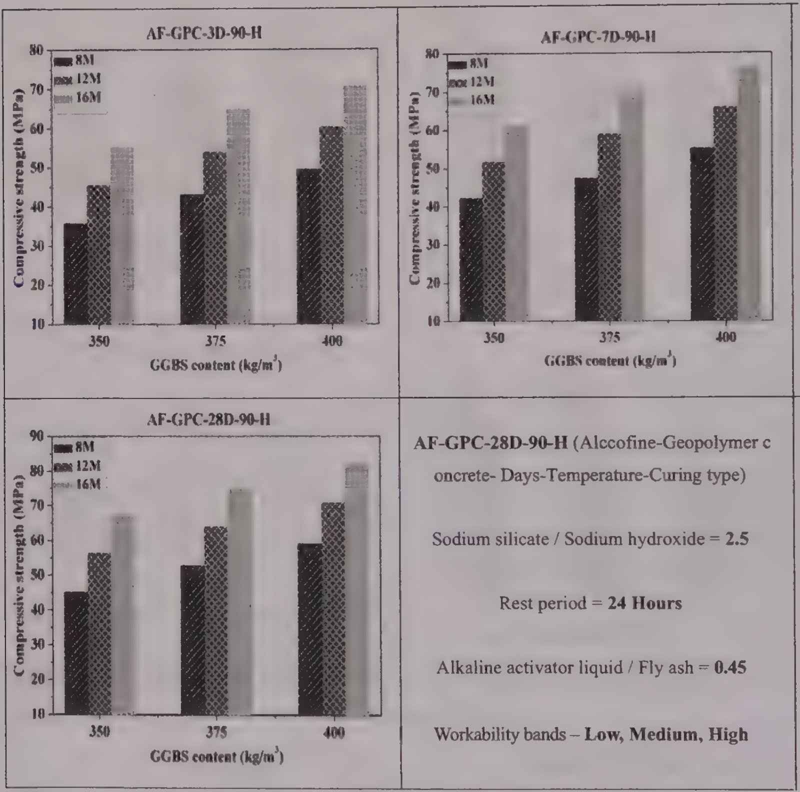
Workability bands – **Low, Medium, High**

Fig. 4.27: Compressive strength with varying GGBS content and curing period at 60^{0}C curing temperature

Effect of GGBS content

It can be observed from Figs. 4.26 to 4.28 that, the compressive strength of the geopolymer concrete increased with the increase in GGBS content into the matrix. For example, when mixes M19GGBSGC to M21GGBSGC were tested after 28 days, a compressive strength of 45.20 MPa, 52.90 MPa and 59.20 MPa was recorded. The percentage increase in compressive strength of heat cured specimens with the increase in GGBS content from 350 kg/m^3 to 375 kg/m^3 and 400 kg/m^3 was 17-18% and 28-29% when 28 days strength was compared. The effect was similar when samples were cured at 60^{0}C. However, at ambient curing, the percentage increase in strength was 39-41% and 71-73%, mixes M1GGBSGC to

M3GGBSGC i.e. with the increase in GGBS content from 350 kg/m^3 to 375 kg/m^3 and 400 kg/m^3 were used for reference. Further, a maximum strength of 82.70 MPa was recorded using 400 kg/m^3 GGBS and at 90^{0}C.



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.28: Compressive strength with varying GGBS content and curing period at 90^{0}C curing temperature

Effect of NaOH molarity

The shape of the strength development curves in Figs. 4.26 to 4.28 indicates that with the increase in NaOH molarity, significant increase in strength can be achieved. For example, when mix M19GGBSGC, M22GGBSGC and M25GGBSGC, cured at 90^{0}C were tested, compressive strength of 45.20 MPa, 56.37 MPa and 67.54 MPa were recorded. It shows that compressive strength increased at higher rate when the molarity increased and at higher curing temperature of 90^{0}C. The percentage increase in compressive strength was 25% and 20% when molarity changes from 8M to 12M and 12M to 16M. The effect was nearly similar when strength of the heat and ambient cured specimens were compared after 28 days.

Effect of curing temperature

It is well known fact in the field of the fly ash based geopolymer concrete that polymerisation process is faster at higher temperature [90]. The similar behaviour was noticed in the geopolymer concrete prepared using GGBS. Further, when heat curing is adopted, 95% of the strength could be achieved after 7 days and the trend was same for all the specimens. Also, when mixes M20GGBSGC, M11GGBSGC and M2GGBSGC were tested, compressive strength of 52.90, 48.65 and 27.20 MPa was noticed. This shows that at elevated heat curing remarkable improvement in strength can be achieved. Also, the behaviour of GGBS based geopolymer concrete was similar to FA and RHA based geopolymer concrete.

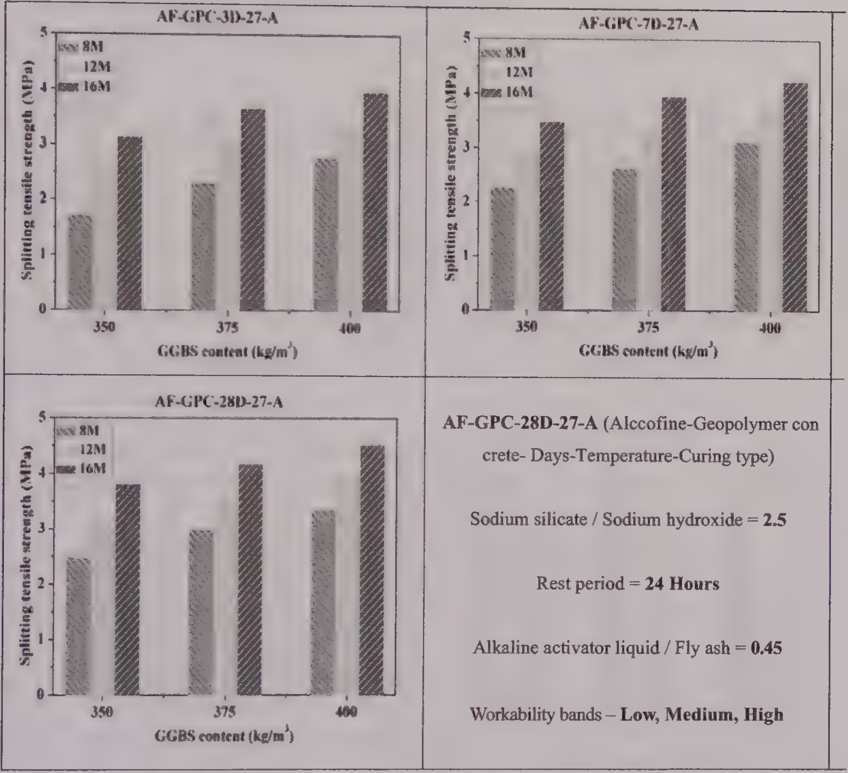
4.3.3.2 Split tensile strength

Similar to compressive strength, split tensile strength was affected by different variables such as NaOH molarities, temperatures and quantity of binder material with the inclusion of alccofine into geopolymer concrete etc. Therefore, five identical samples for each mix were cast, cured at 27° C, 60° C and 90° C and tested at the age of 3, 7 and 28 days to study their effects on the split tensile strength. The average of five specimens is reported in the Table 4.11. Figs. 4.29 to 4.31 shows the relationship between compressive strength and GGBS content along with different variables.

Table 4. 11: Split tensile strength of GGBS based GPC

Mixture	**Average split tensile strength (MPa)**		
	3 days	**7 days**	**28 days**
M1GGBSGC	1.71	2.26	2.47
M2GGBSGC	2.31	2.62	2.98
M3GGBSGC	2.77	3.12	3.35
M4GGBSGC	2.50	2.91	3.19
M5GGBSGC	3.05	3.34	3.62
M6GGBSGC	3.41	3.71	3.97
M7GGBSGC	3.13	3.47	3.80
M8GGBSGC	3.66	3.95	4.17

M9GGBSGC	3.97	4.24	4.52
M10GGBSGC	3.24	3.60	3.75
M11GGBSGC	3.64	3.86	4.14
M12GGBSGC	3.97	4.24	4.43
M13GGBSGC	3.79	4.10	4.32
M14GGBSGC	4.20	4.43	4.66
M15GGBSGC	4.49	4.74	4.95
M16GGBSGC	4.27	4.54	4.81
M17GGBSGC	4.70	4.93	5.12
M18GGBSGC	4.95	5.18	5.42
M19GGBSGC	3.48	3.82	3.97
M20GGBSGC	3.86	4.07	4.33
M21GGBSGC	4.18	4.44	4.62
M22GGBSGC	3.99	4.28	4.49
M23GGBSGC	4.38	4.60	4.82
M24GGBSGC	4.66	4.90	5.11
M25GGBSGC	4.45	4.71	4.97
M26GGBSGC	4.86	5.09	5.28
M27GGBSGC	5.11	5.34	5.57



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

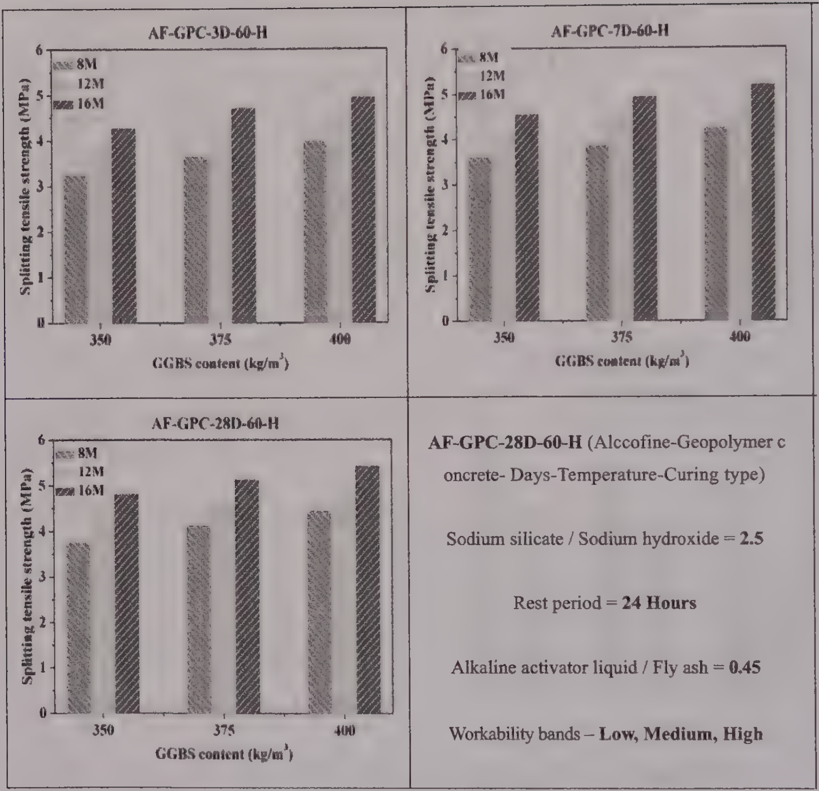
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.29: Split tensile strength with varying GGBS content and curing period at 27°C curing temperature



AF-GPC-28D-60-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

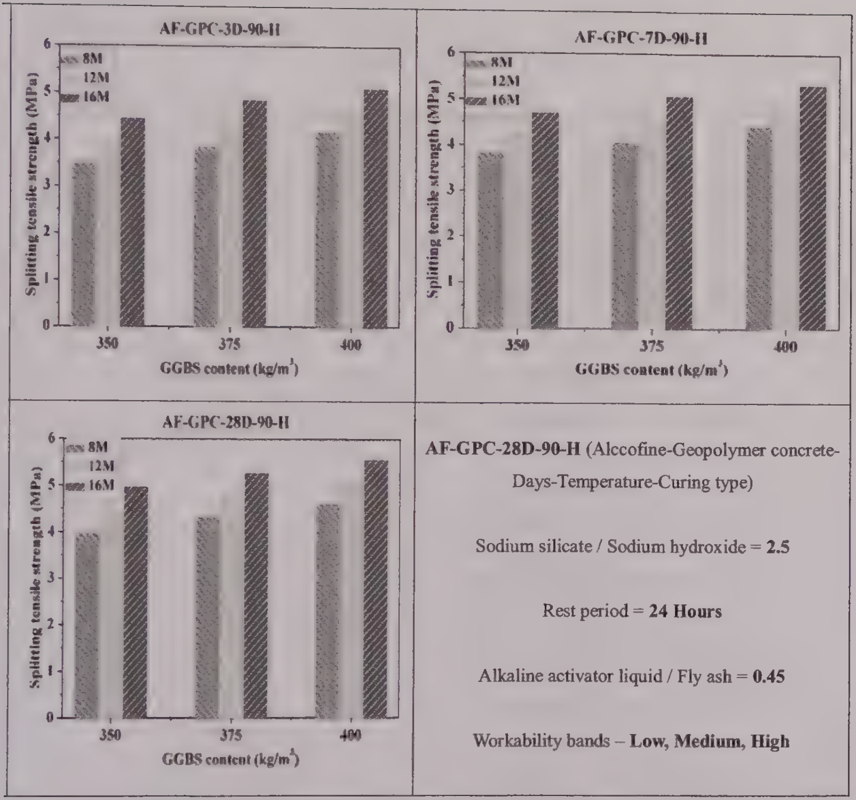
Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.30: Split tensile strength with varying GGBS content and curing period at 60^{0}C curing temperature

Increase in split tensile strength was noticed as concentration of NaOH i.e. molarities, GGBS content and curing temperatures were increased. For example, the increase in strength was about 10-12% when molarity changed from 8M to 12M (M21GGBSGC & M24GGBSGC) and about 20-22% when 8M to 16M (M21GGBSGC & M27GGBSGC) for the same GGBS content and at the age of 28 days.



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete-Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.31: Split tensile strength with varying GGBS content and curing period at 90^{0}C curing temperature

As discussed above, curing temperature played an important role and significantly affected the strength of the geopolymer concrete. The same was noticed here in case of split tensile strength. It was observed that with the increase in the temperature the split tensile strength increased.

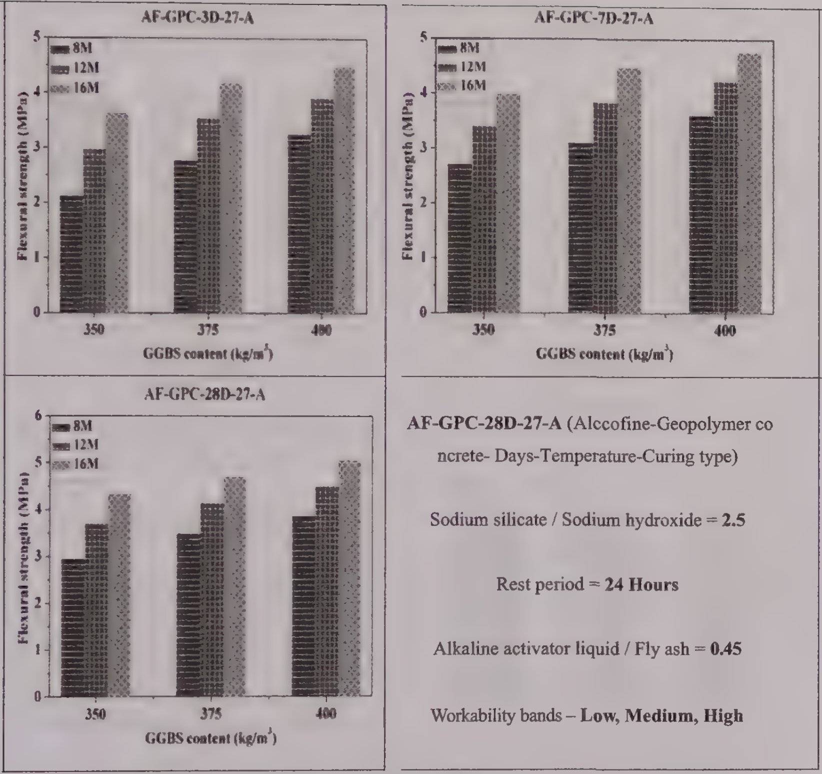
4.3.3.3 Flexural strength

Specimens for measuring the flexural strength in terms of modulus of rupture were cast and tested in the similar way as discussed for FA and RHA based GPC. Flexural strength in the

Table 4.12 is average over three specimens. Figs. 4.32 to 4.34 show the flexural strength for geopolymer concrete specimens at 27° C, 60° C and 90° C.

Table 4. 12: Flexural strength of GGBS based GPC

Mixture	Average flexural strength (MPa)		
	3 days	7 days	28 days
M1GGBSGC	2.13	2.72	2.95
M2GGBSGC	2.78	3.11	3.49
M3GGBSGC	3.27	3.63	3.87
M4GGBSGC	2.98	3.41	3.70
M5GGBSGC	3.55	3.85	4.14
M6GGBSGC	3.93	4.24	4.50
M7GGBSGC	3.64	3.99	4.33
M8GGBSGC	4.19	4.48	4.70
M9GGBSGC	4.50	4.77	5.05
M10GGBSGC	3.76	4.12	4.28
M11GGBSGC	4.16	4.39	4.67
M12GGBSGC	4.50	4.77	4.96
M13GGBSGC	4.32	4.63	4.85
M14GGBSGC	4.73	4.96	5.19
M15GGBSGC	5.02	5.27	5.48
M16GGBSGC	4.80	5.07	5.34
M17GGBSGC	5.22	5.46	5.65
M18GGBSGC	5.48	5.70	5.94
M19GGBSGC	4.00	4.35	4.50
M20GGBSGC	4.38	4.60	4.87
M21GGBSGC	4.71	4.97	5.15
M22GGBSGC	4.52	4.81	5.02
M23GGBSGC	4.91	5.13	5.35
M24GGBSGC	5.19	5.43	5.64
M25GGBSGC	4.98	5.24	5.50
M26GGBSGC	5.39	5.62	5.80
M27GGBSGC	5.64	5.85	6.08



AF-GPC-28D-27-A (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

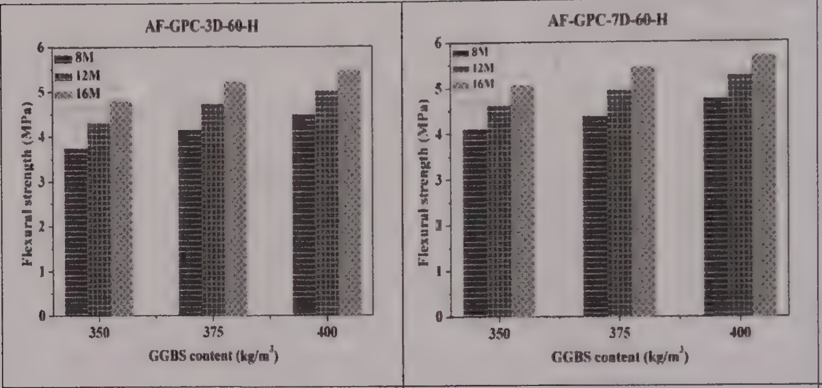
Sodium silicate / Sodium hydroxide = **2.5**

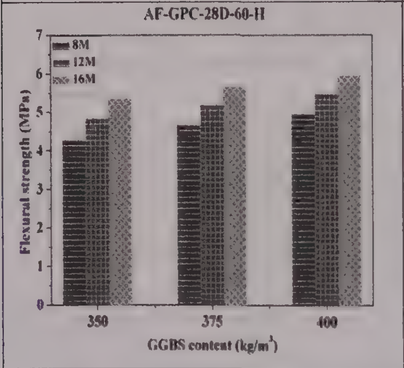
Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.32: Flexural strength with varying GGBS content and curing period at $27^{O}C$ curing temperature





AF-GPC-28D-60-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

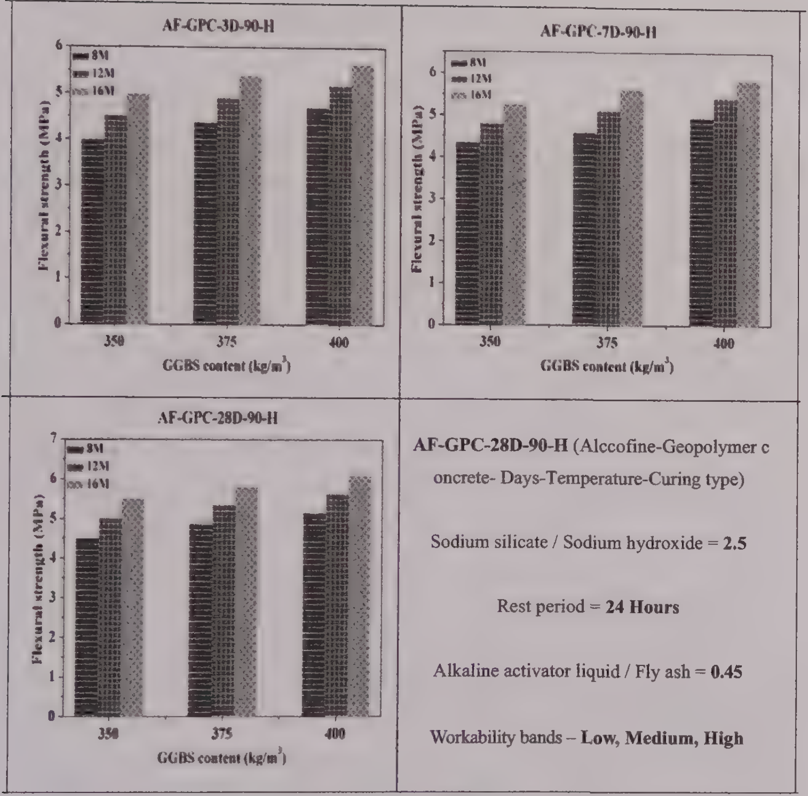
Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.33: Flexural strength with varying GGBS content and curing period at $60^{O}C$ curing temperature



AF-GPC-28D-90-H (Alccofine-Geopolymer concrete- Days-Temperature-Curing type)

Sodium silicate / Sodium hydroxide = **2.5**

Rest period = **24 Hours**

Alkaline activator liquid / Fly ash = **0.45**

Workability bands – **Low, Medium, High**

Fig. 4.34: Flexural strength with varying GGBS content and curing period at 90^{O}C curing temperature

Flexural strength of the geopolymer specimens had similar effects as were observed for split tensile strength. Moreover, the effects of various parameters such as temperature, molarity and binder content on the strength of GGBS based GPC were similar to those obtained for FA and RHA based geopolymer concrete. For example, flexural strength of ambiently (M7GGBSGC to M9GGBSGC) and heat cured (M25GGBSGC to M27GGBSGC) specimens at the age of 28 days were 4.33, 4.70 and 5.05 & 5.50, 5.80 and 6.08 MPa were obtained. This showed that strength increased significantly with the increase in GGBS content and temperature. Although the strength of the GGBS based geopolymer concrete was higher than

FA and RHA based GPC for each mix yet ultimate strength was obtained for the specimen having 400kg GGBS, 16M and at heat curing.

Summary:- This Chapter has provided the details of the results of the structural properties like compressive, split tensile and flexural strengths of geopolymer concrete. Therefore, it can be concluded from the above results of compressive, split and flexural strengths of the geopolymer concrete prepared with fly ash, rice husk ash, ground granulated blast furnace slag that it is suitable for precast and cast in situ works.

CHAPTER - 5

ANALYSIS OF THE RESULTS

5.1 INTRODUCTION

This chapter includes the analysis of the strength results which were obtained during the testing and have been presented in the Chapter 4. Further, based upon the design aids and results which have been discussed in the Chapter-4, a method has been proposed in this chapter for the purpose of developing mix design method for geopolymer concrete which satisfied the requirements of target strength and workability. For the mixes tabulated in Tables 3.12 to 3.14 in Chapter-3, analysis has been done by using the design aids (AF-GPC graphs, Figs. 4.8 to 4.10, Figs. 4.17 to 4.19 and Figs. 4.26 to 4.28). Further, this chapter includes the validation of the proposed methodology, which has been done with the help of the examples. In addition, structural behaviour of the GPC has been studied and discussed at the end of this Chapter, which covers the stress-strain behaviour, relationships between structural properties and comparison of the proposed relationships with different design standards to check the applicability of the GPC into industrial

5.2 ANALYSIS ON THE STRENGTH RESULTS

The rate of increase in strength with age in the case of fly ash and RHA based GPC was similar to conventional concrete [35, 38, 147, 148] when specimens were ambiently cured. The ambiently cured, slag based GPC specimens possessed significantly higher early strength (45MPa) than FA (15MPa) and RHA(13MPa) based specimens, it may be due combination of polymerisation and hydration in the presence of CaO. Further, GGBS is the main source of CaO and silica, and addition of alccofine further increases the CaO content into the matrix, this could have been the reason of high strength at ambient temperature in the case of GGBS based geopolymer concrete.

Also, early age strength was increased significantly for all the cases when the geopolymer specimens were cured at heat temperature. This meant that geopolymer concrete could be considered as beneficial for the construction applications where high early strength is required. The increased early age strength was due to the formation of calcium silicate hydrate (CSH) and polymeric products CASH and sodium aluminate silicate hydrate (NASH) formed due to increased rate of polymerisation due to alccofine. Also, the calcium present in the alccofine reacted with the alkaline activators and produced heat which might have helped in increasing the temperature of curing internally for geopolymers [41, 145, 149] and thus enhanced its compressive, split and flexural strengths. The formation of these products has also been confirmed in XRD and SEM studies as discussed in Chapter 3. Fly ash is the main source of the silica and alumina, Rice husk ash is the main source of silica and as discussed above GGBS is the main source of CaO and silica, which increases when the amount of raw materials increased in the system and it influence the polymerisation reaction and hence increase the NASH and CASH which results into higher strength.

Also, due to addition of alccofine, CSH formed and requires water curing to enhance the final age strength of the mix which was not provided. Hence water curing may have increased the strength of the geopolymer specimens as in the case of the conventional concrete. Further, the additional water that was added while preparing the mixes, could have reacted with calcium to form CSH into the matrix which lead heat of hydration and corresponding higher strength. Previous studies also showed that enhancement of the mechanical properties by increasing the calcium content in the system was limited to a certain extent only and after that, it showed negative effects as water curing is not preferred in geopolymer concrete [90, 150-152]. Further, geopolymer mechanism involves polymeric reactions that initiate with high temperature curing so bulk of the strength gets achieved at an early stage when heat curing adopted instead of ambient temperature. The combine network of the CSH and polymeric reactions was expected to have dense micro-structure after 28 days of ambient curing and after 7 days of heat curing which therefore increased the mechanical properties of the geopolymer concrete and the same has also been confirmed during the preliminary laboratory test results.

5.3 AF-GPC GRAPHS

AF-GPC-Graphs (Alccofine, Geopolymer-Graphs) have been developed using the experimental data given in the Tables 3.12 to 3.14 for the investigation and these graphs lie at the origin of the developed design mix process. These graphs (Figs. 4.8 to 4.10, Figs. 4.17 to 4.19 and Figs. 4.26 to 4.28) originally represent the relationship between the quantum of raw material (fly ash, rice husk ash and ground granulated blast furnace slag) and compressive strength at different curing conditions and molarities. Compressive strength results obtained for the mixes tabulated in Tables 4.2 to 4.4 were used to develop the proposed AF-GPC-Graphs. Further, AF-GPC graphs have been proposed for 3, 7 and 28 days compressive strength. To choose the exact type of mix, workability bands have been provided in the Table 5.1 for different raw material quantities. The workability of the fresh GPC was measured using the standard slump [142]. The classification of the slump values was done as per the condition of compaction and using the clause 7 of IS 456:2000 [153]. Based on the different slump values GPC was classified in terms of a very highly workable, highly workable, medium workable and low workable [142, 153]. Workability on the similar grounds has also been fixed by British standards and American concrete institute. Slump values observed for the mixes tabulated in Tables 4.2 to 4.4 are plotted and have been shown with help of the Fig. 4.8, Fig. 4.11 and Fig. 4.13. Further, the above-mentioned criterion has been related to the quantum of raw material (fly ash, rice husk ash and ground granulated blast furnace slag) as shown in Table 5.1.

Table 5. 1: Workability bands used for geopolymer concrete

Raw material [RM] Kg/cum	**RM<350**	**350< RM<375**	**375< RM<400**	**RM>400**
Degree of workability/Slump	Less [<75]	Medium [>75 but <100]	High [>100 but <150]	Very High [>150]

*RM- fly ash or rice husk ash or ground granulated blast furnace slag

5.4 PROPOSED METHOD FOR DESIGNING GPC

An attempt has been made, to propose mix design methodology for alccofine added GPC in a rational way. By fixing the certain parameters in the production of GPC, it can be made economical and flexibility can be rendered in the design mixes, in view of strength requirement and desired activator solution. The flexibility is in terms of requirements of compressive strength and workability with important parameters of GPC such as its amount, curing temperature and concentration of solution. The design procedure of the proposed mix design has been outlined in the form of flow chart as stated in Fig. 5.1 and the step by step procedure is summarized as follows;

Firstly, a target compressive strength and workability depending on structural requirements and size of the structural member are set for the design process. This target strength is then established on the AF-GPC-Graphs (Figs. 4.15 to 4.17, Figs. 4.27 to 4.29 and Figs. 4.39 to 4.41), from where corresponding values of raw material (Fly ash or RHA or GGBS) content, molarity of NaOH solution and curing temperature are obtained. The quantum is also re-examined through Table 5.1 for the requirement of workability. Higher of the two values of the raw material is finalised. However, if workability is not achieved for the located raw material quantum and target strength using the proposed method, then amount of superplasticizer can be increased to reach at the required degree of workability level. The AF-

GPC-Graphs presented in this study shows relationship between compressive strength and raw materials. However, this relationship has been established at 2% and 10% (by weight of raw material) superplasticizer and alccofine, respectively.

As discussed in the Chapter 2 with reasons that alkaline activator liquid (AAL) to fly ash (FA) ratio and sodium silicate to sodium hydroxide ratio were selected 0.45 and 2.5, respectively. The next step in the mix design of GPC is to calculate the value of NaOH and Na_2SiO_3 solutions by using the following equations

$$\frac{\text{AAL}}{\text{FA}} = \frac{\text{Sodium Silicate solution} + \text{Sodium hydroxide solution}}{\text{Fly Ash}} = 0.45 \dots\dots\dots (5.1)$$

From which; AAL = 0.45 x Fly ash

$$\frac{\text{Sodium Silicate}}{\text{Sodium hydroxide}} = 2.5 \dots\dots\dots (5.2)$$

From which; NaOH solution = AAL/3.5 and Na_2SiO_3 solution = 2.5 x sodium hydroxide solution

Water (sum of masses of additional free water and water used while preparing Na_2SiO_3 and NaOH) to geopolymer binder (sum of masses of fly ash, alccofine, NaOH solids and Na_2SiO_3 solids) ratio (W/GPB) has been kept 0.27 for this study [31, 36, 51, 154-157]. Knowing the above values, amount of water and solids used in alkaline activator solution [AAL] can be calculated accurately. Additional free water quantity can be calculated using the Eq. [5.3].

$$\frac{\text{W}}{\text{GPB}} = \frac{\text{WOH} + \text{WSI} + \text{Wextra}}{\text{AF} + \text{FA} + \text{Solids NaOH} + \text{Solids Na2SiO3}} = 0.27 \dots\dots\dots (5.3)$$

Where, WOH, WSI, W_{extra} is the water present in the hydroxide solution, silicate solution and any additional free water in the system, respectively. AF, FA, Solids NaOH, and Solids Na_2SiO_3, are the alccofine, fly ash and solids present in the NaOH solution, silicate solution, respectively. In this study, the density of the GPC has been considered as 2350 kg/m^3 on experimental basis and 70-75% of the mass of the GPC has been made up for the saturated surface dry aggregates. The aggregates used are well graded through 14, 10 and 7mm in size and in the proportion of 45, 35 and 20%, respectively. Further, as discussed in the Chapter 2 maximum compressive strength has been obtained by taking the ratio of coarse to fine aggregates equals to 70:30 out of the total aggregates, same was considered during this study.

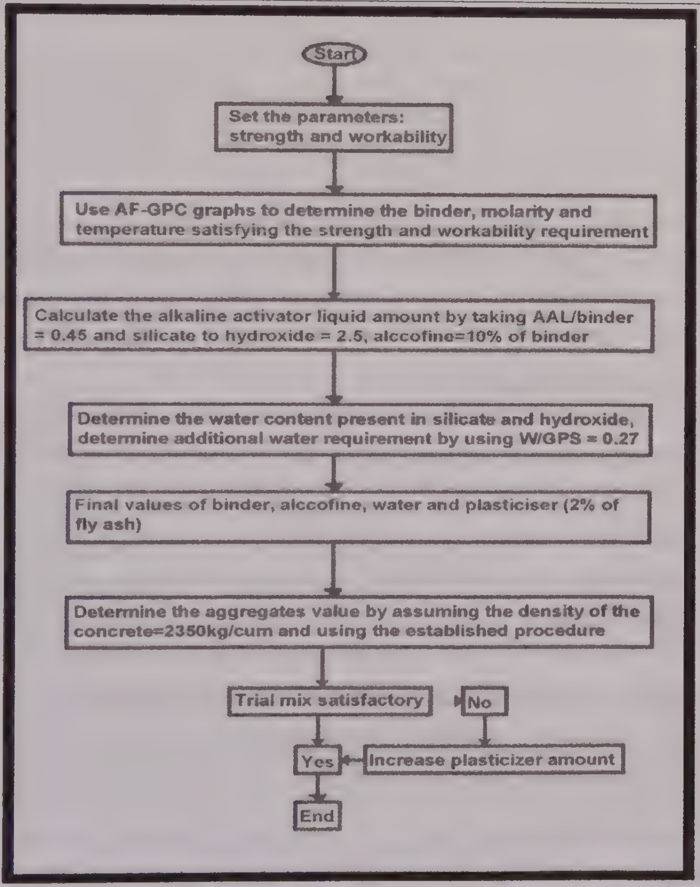


Fig. 5.1: Flow chart of the proposed mix design process for alccofine based GPC

5.4.1 Proposed method for sample preparation

Different researchers have tried different methods for the sample preparation of the geopolymer concrete and suggested that the compressive strength of the GPC was not affected by mixing. The procedure suggested for the preparation of the GPC samples is as discussed below [157]:

1. NaOH was prepared before 24 hours of the casting and uniformly mixed with the Na_2SiO_3 1hr prior to the mixing of the ingredients of the GPC.
2. All the dry ingredients of GPC mixture were then mixed for at least 5min in the Pan mixture followed by liquid ingredient, then poured into 150 mm size standard moulds and compaction was done on a vibrating Table for about 2-3 minutes. Sealed samples were then placed at room temperature for 24 hours rest period.
3. The samples were then heat cured at 60° and 90° C for 24 hours in an electric oven after a rest period of one day. After heat curing, the samples were returned to room temperature till the time of testing. However, the ambient cured samples were kept at the room temperature (27° C) till the time of testing.

5.4.2 Limitations of the proposed method

Efforts were made to achieve the target compressive strength and workability in a rational and most economical way. However, this method has certain limitations which are as discussed below:

1. Ratios of raw material to alkaline activator liquid and water to geopolymer binders used in this study are 0.45 and 0.27, respectively with the reasons detailed in the Chapter 2 and 3.
2. Coarse aggregate to fine aggregate ratio should be 70:30 out of the total aggregates.

5.5 VERIFICATION OF THE MIX METHODOLOGY USING AN EXAMPLE

To validate the proposed method, an example was undertaken here to design the mix of GPC at heat and ambient curing for ordinary (cast-in-situ) and precast members.

5.5.1 Fly ash based concrete

Assuming, the required compressive strength and workability for ordinary and precast GPC members is 25MPa (Trial mix – TM-25), 75mm and 35MPa (Trial mix -TM-35), 75mm, respectively. The first step is to calculate the target mean strength. For this study, the target mean strength has been calculated as per Indian standard [158] and equals to $1.65 \times S + F_{ck} = F_{ck'}$ i.e. 31.6 for TM25 and 43.25 for TM35, where $F_{ck'}$ is target mean compressive strength, F_{ck} is characteristics compressive strength (28 days for ambient curing and 7 days for heat curing), S is standard deviation. Several NaOH molarity and curing temperatures combinations could be used to achieve this target strength which are presented in AF-GPC-Graphs and Table 5.1 can be used to target the required workability. Nevertheless, and for the sake of illustration here, the AF-GPC-Graph (Fig. 4.8) is chosen and calculations are illustrated for mix TM-25. Fly ash quantity can be chosen from the AF-GPC-28D-27-A Graph for the required target mean strength and from Table 5 whichever is more i.e. 375 kg/m^3, using this the corresponding values of alccofine, NaOH, Na_2SiO_3, superplasticizer and aggregates can be calculated and are given below.

Alkaline activator liquid (AAL) is calculated using Eq. 5.1:

$0.45 \times 375 = 168.75\ kg/m^3$.

Alccofine = $0.10 \times 375 = 37.5$ kg

Using Eq. 1 and Eq. 2 the values of the NaOH and Na_2SiO_3 can be calculated.

NaOH solution = $168.75/3.5 = 48.21$ kg

Na_2SiO_3 solution = $2.5 \times 48.21 = 120.53$ kg

The mass of solids and water present in the NaOH and Na_2SiO_3 can be calculated by using the composition of NaOH [40.4% solids by weight for 16M] and Na_2SiO_3 [44.1% solids by weight as per supplier's specifications] by weight and is given below.

Mass of solids in NaOH = $(40.4/100) \times 48.21 = 19.48$ kg

Mass of water in NaOH = $48.21-19.48= 28.73$ kg

Mass of solids in Na_2SiO_3 = $(44.1/100) \times 120.53 = 53.15$ kg

Mass of water in Na_2SiO_3 = $120.53-53.15 = 67.38$ kg

Moreover, the AF-GPC-Graphs have been developed for W/GPB equals to 0.27. Thus, extra water quantity can be calculated using the Eq. 5.3.

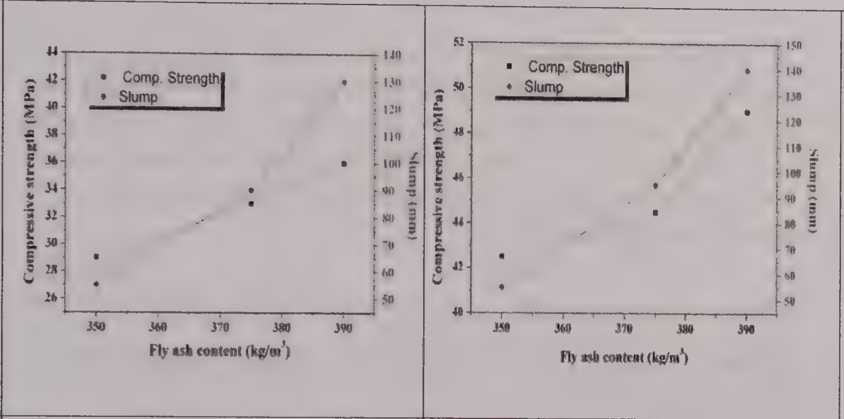
$(W_{extra} + 28.73 + 67.38) / (37.5 + 375 + 19.48 + 53.15) = 0.27$; from which $W_{extra} = 34.87$ kg.

By using the volumetric analysis, the other parameters like total aggregate can be calculated. Generally, in OPC based concrete the fine aggregate to coarse aggregate ratio is 30–70 and the same has been considered here. Commercially available 2% high dosage superplasticizer is added and the percentage of superplasticizer is slightly altered to satisfy the workability requirements, if needed. Similarly, on the same ground mix TM-35 have been designed. In addition, two more trial mixes were made with the target compressive strength of 25, 35 and 40, 50 for TM-25 and TM-35 mix, respectively, to make the geopolymer concrete economical. The same practice of casting three trial mixes is also followed by varying the water to cement ratio while mix designing the conventional concrete in the laboratory for actual and accurate determination of ingredients quantities depending on their properties. The final values of the materials and the strength achieved using the above proposed method are listed in Table 5.2 below for mixes TM-25 and TM-35 (TM-25: trial mix- target strength in MPa).

Table 5. 2: Mix design quantities (kg) carried out using the above method for one cubic meter.

Quantities	TM-25	TM-25	TM-25	TM-35	TM-35	TM-35
Target strength [MPa]	25	31.6	35	40	43.25	50
Required slump [mm]	75	75	75	75	75	75
Fly ash	350	375	390	350	375	390
Alccofine	35	37.5	39	35	37.5	39
Coarse Aggregate	1236	1207	1191	1239	1200	1193
Fine aggregate	530	517	510	532	514	512
NaOH	45	48.21	50.14	48.21	51.42	50.14
Molarity[M]	16	16	16	12	12	12
Na_2SiO_3	112.50	120.53	125.25	120.53	128.57	125.35
Extra water	34.83	37.32	38.81	30.08	32.23	33.52
Plasticizer	7	7.5	7.8	7	7.5	7.8
Curing	Ambient [28 Days] @ 27°C	Ambient [28 Days] @ 27°C	Ambient [28 Days] @ 27°C	Heat [24 Hours] @ 90°C	Heat [24 Hours] @ 90°C	Heat [24 Hours] @ 90°C
Average strength [MPa]	30.22	37.78	41.33	45.33	46.66	51.55
Slump [mm]	55	90	130	55	95	140
Testing	28 Days			7 Days		

Based upon the above values of the ingredients as shown in Table 5.2, 30 cubes for every trial mix were cast and tested. The average strength in the Table 5.2 is average above 30 cubes. Fig. 5.2 shows the relationship between compressive strength and slump values with various fly ash contents achieved from the Table. 5.2 for the trial mixes of TM-25 and TM-35.



(a) TM-25 *(b) TM-35*

Fig. 5.2: Compressive strength & Slump and various fly ash content

By referring the Fig. 5.2, final recommended values of the different ingredients of the fly ash based GPC for TM-25 and TM-35 could be as given in Table 5.3.

Table 5. 3: Recommended quantities (kg) for the mix TM-25 and TM-35.

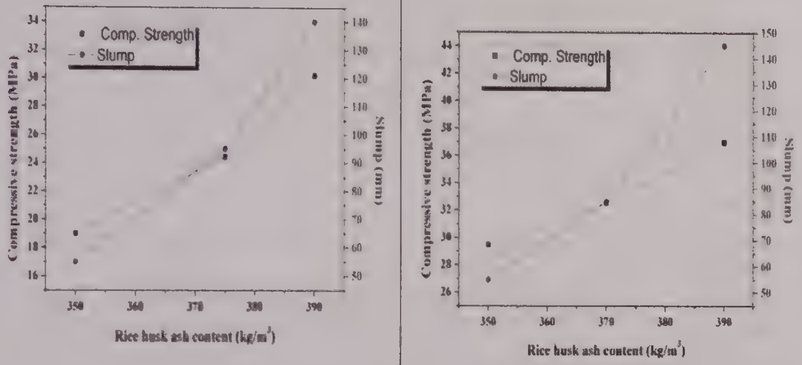
Quantities	TM-25	TM-35
Fly ash	365	365
Alccofine	36.5	36.5
Coarse Aggregate	1218	1222
Fine aggregate	522	524
NaOH	46.9	46.9
Molarity[M]	16	12
Na_2SiO_3	117.3	117.3
Extra water	36.3	31.4
Plastisizer	7.3	7.3
Curing	Ambient [28 Days] @ 27°C	Heat [24 Hours] @ 90°C
Testing	28 Days	7 Days

5.5.2 Rice husk ash based concrete

To validate the proposed method for mix design using RHA as a binder material, an example is undertaken here to design the mix of GPC at heat and ambient curing on the same ground as discussed above. The final values of the materials and the strength achieved using the above proposed method are listed in Table 5.4 below for mix TM-15 and TM-25 (TM-15: trial mix- target strength in MPa).

Table 5. 4: Mix design quantities (kg) carried out using the above method for one cubic meter.

Quantities	TM-15	TM-15	TM-15	TM-25	TM-25	TM-25
Target strength [MPa]	15	20.775	25	25	31.6	35
Required slump [mm]	75	75	75	75	75	75
RHA	350	375	400	350	370	400
Alccofine	35.0	37.5	40.0	35.0	37.0	40.0
Coarse Aggregate	1243	1215	1186	1243	1220	1186
Fine aggregate	533	521	508	533	523	508
NaOH	45.0	48.2	51.4	45.0	47.6	51.4
Molarity[M]	8	8	8	8	8	8
Na_2SiO_3	112.5	120.5	128.6	112.5	118.9	128.6
Extra water	24.4	26.2	27.9	24.4	25.8	27.9
Plastisizer	7.0	7.5	8.0	7.0	7.4	8.0
Curing	Ambient [28 Days] @ 27°C	Ambient [28 Days] @ 27°C	Ambient [28 Days] @ 27°C	Heat [24 Hours] @ 90°C	Heat [24 Hours] @ 90°C	Heat [24 Hours] @ 90°C
Average strength [MPa]	19.00	24.45	30.22	29.45	32.55	37.00
Slump [mm]	55	95	140	55	85	145
Testing	28 Days			7 Days		



(a) TM-15

(b) TM-25

Fig. 5.3: Compressive strength & Slump and various RHA content

Fig. 5.3 shows the relationship between compressive strength & slump values with various fly ash content achieved from the Table. 5.4 for both the mixes, TM-15 and TM-25. By referring the Fig. 5.3, final values of the rice husk ash content for TM-15 and TM-25 can be calculated and it was in between 350 to 360 kg/cum for TM-15 and in between 370 to 375 kg/cum for TM-25, which will target the slump and compressive strength. The final recommended values for TM-15 and TM-25 of RHA based GPC could be as given in Table 5.5.

Table 5. 5: Recommended quantities (kg) for the mix TM-15 and TM-25.

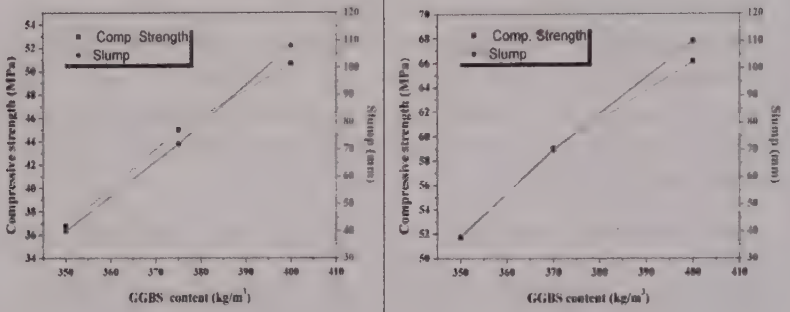
Quantities	TM-15	TM-25
RHA	357	373
Alccofine	35.7	37.3
Coarse Aggregate	1235	1217
Fine aggregate	529	522
NaOH	45.9	48.0
Molarity[M]	8	8
Na_2SiO_3	114.8	119.9
Extra water	24.9	26.0
Plastisizer	7.14	7.46
Curing	Ambient [28 Days] @ 27°C	Heat [24 Hours] @ 90°C
Testing	28 Days	7 Days

5.5.3 GGBS based concrete

The proposed methodology has also been applied to slag based geopolymer concrete to design the trial mixes for 35MPa and 50MPa strength concrete. Similar to fly ash and RHA based concrete, an example is undertaken here to design the mix of GPC at heat and ambient curing. The final values of the materials and the strength achieved using the above proposed method are listed in Table 5.6 below for mix TM-35 and TM-50 (TM-50: trial mix- target strength in MPa).

Table 5. 6: Mix design quantities (kg) carried out using the above method for one cubic meter.

Quantities	**TM-35**	**TM-35**	**TM-35**	**TM-50**	**TM-50**	**TM-50**
Target strength [MPa]	35	43.25	50	50	58.25	65
Required slump [mm]	75	75	75	75	75	75
GGBS	350	375	400	350	375	400
Alccofine	35.0	37.5	40.0	35.0	37.5	40.0
Coarse Aggregate	1236	1207	1178	1239	1210	1181
Fine aggregate	530	517	505	531	519	506
NaOH	45.0	48.2	51.4	45.0	48.2	51.4
Molarity[M]	16	16	16	12	12	12
Na_2SiO_3	112.5	120.5	128.6	112.5	120.5	128.6
Extra water	34.9	37.3	39.8	30.1	32.2	34.4
Plastisizer	7.0	7.5	8.0	7.0	7.5	8.0
Curing	Ambient [7 Days] @ 27°C	Ambient [7 Days] @ 27°C	Ambient [7 Days] @ 27°C	Heat [24 Hours] @ 90°C	Heat [24 Hours] @ 90°C	Heat [24 Hours] @ 90°C
Average strength [MPa]	36.75	45.00	50.65	51.70	59.00	66.10
Slump [mm]	40	72	108	38	70	110
Testing	7 Days			7 Days		



(a) TM-35 *(b) TM-50*

Fig. 5.4: Compressive strength & Slump and various GGBS content

Fig. 5.4 shows the relationship between compressive strength & slump values with various GGBS content achieved from the Table. 5.6 for both the mixes, TM-35 and TM-50, respectively. By referring the Fig. 5.4, final values of the GGBS content for TM-35 and TM-50 could be recommended, which will target the slump and compressive strength. The same have been shown in Table 5.7.

Table 5. 7: Recommended quantities (kg) for the mix TM-25 and TM-35.

Quantities	TM-35	TM-50
GGBS	375	375
Alccofine	37.5	37.5
Coarse Aggregate	1207	1210
Fine aggregate	517	519
NaOH	48.2	48.2
Molarity[M]	16	12
Na_2SiO_3	120.5	120.5
Extra water	37.3	32.2
Plastisizer	7.5	7.5
Curing	Ambient [7 Days] @ 27°C	Heat [24 Hours] @ 90°C

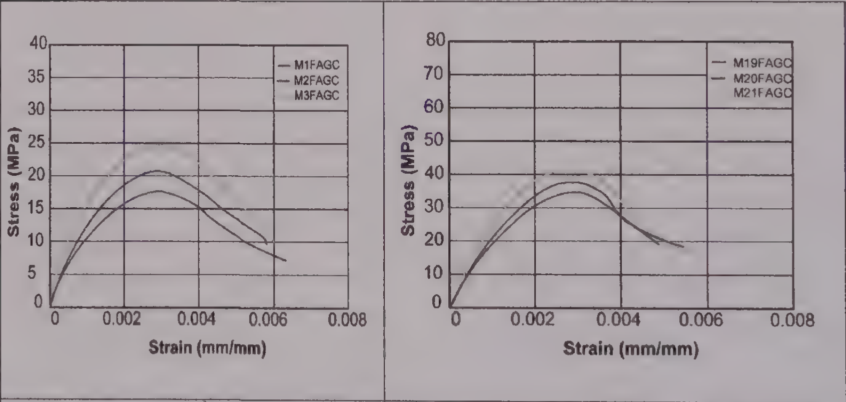
Therefore, the mix design method which has been proposed and validated above with the help of the examples is suitable for designing the mix for geopolymer concrete. Using the proposed method, geopolymer concrete of grades ranging from 10MPa to 70MPa can be designed using different alumino-silicate materials like fly ash, RHA and GGBS and design aids. Further, the proposed methodology can also be used to develop the geopolymer concrete at ambient temperature and the same has been validated above. The above validation confirms that the required workability while in fresh state and strength in hardened state can be achieved at high and ambient temperature, respectively.

5.6 STRUCTURAL BEHAVIOUR OF GPC

Structural behaviour of the geopolymer concrete containing different alumino-silicate materials in terms of stress-strain behaviour, compressive, split tensile and flexural strengths, and elastic modulus have been studied and discussed here. To study the exact behaviour under compression three cylinders were cast and tested for each mix. Tests were done on the open loop Universal testing machine under compression. Further, to compare the results obtained for GPC during this study with conventional concrete, reference of the study by Aitcin and Mehta (1990) and Warner et al. (1998) have been made. Previous studies on the conventional and fly ash based geopolymer concrete show that strain at the peak stress was in the range of 0.002-0.0038 [125, 159-162] and slope was on higher side after the peak stress when compared with the slope prior to peak stress which show brittleness of the material. In addition random mixes of the fly ash based GPC were taken to compare the stress strain behaviour of the geopolymer concrete specimens with the model proposed by the Junaid et al.[162] in the end of this Chapter.

5.6.1 Stress-strain behaviour of fly ash based GPC

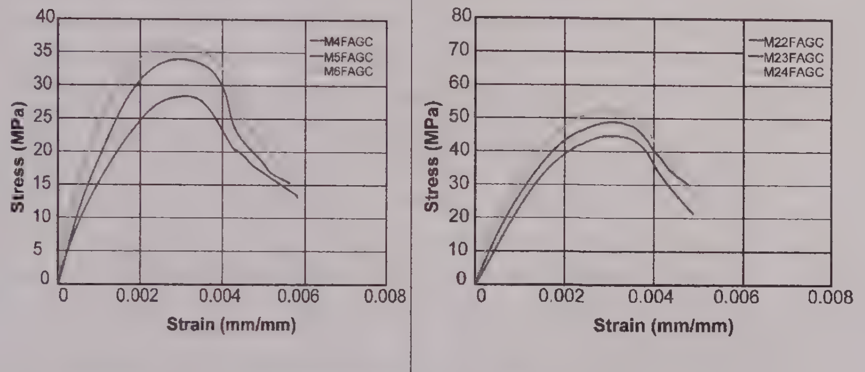
Tests were done on the geopolymer concrete specimens prepared with different molarity and curing temperature, the curve were drawn and shown in Figs. 5.5 to 5.7.



(a) Curing period at 27^0 C curing temperature

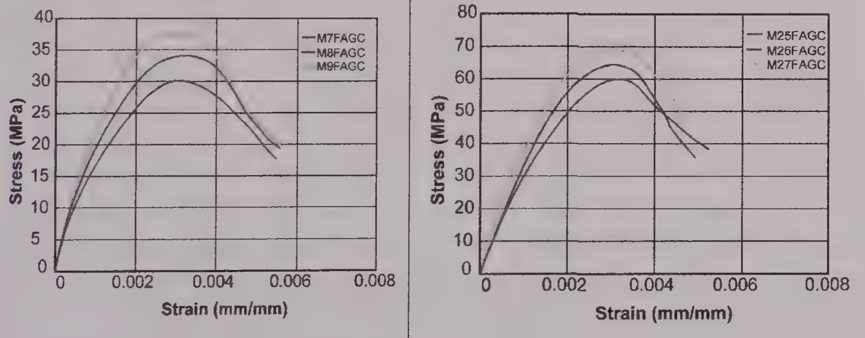
(b) Curing period at 90^0 C curing temperature

Fig. 5.5: Stress strain curves of GPC specimens using 8M NaOH



(a) Curing period at 27⁰ C curing temperature *(b) Curing period at 90⁰ C curing temperature*

Fig. 5.6: Stress strain curves of GPC specimens using 12M NaOH



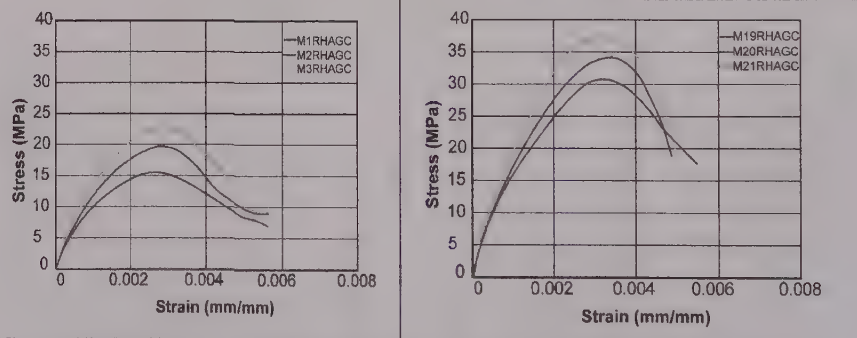
(a) Curing period at 27⁰ C curing temperature *(b) Curing period at 90⁰ C curing temperature*

Fig. 5.7: Stress strain curves of GPC specimens using 16M NaOH

The stress strain curves were obtained directly on the Universal Testing Machine under compression. Figs. 5.5 to 5.7 show the average stress strain behaviour of three specimens. The rate of strain increase at the initial stage, was slower and the trend continued upto 80% of the maximum stress. Further, the rate of increase in strain of geopolymer concrete was a little bit faster than conventional concrete [31] and this might be due to large number of micro cracks developed near peak stress as observed by other researchers [40, 125, 163]. Further, geopolymer concrete have brittle failure and it was noticed same for all the geopolymer concrete specimens. Also, the maximum strain (ε_o) was observed to be in the range of 2.5-3.5 x 10^{-3} mm/mm, at maximum stress in all the cases as shown in above Figs. The peak point position in the stress-strain curves was influenced by compressive strength while, rate of straining and loading were kept constant.

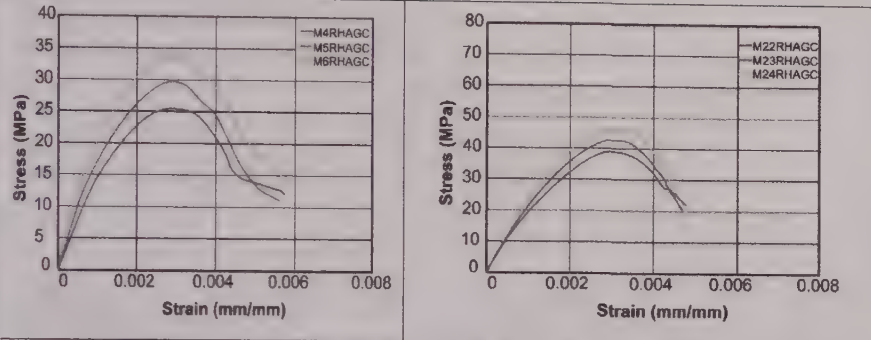
5.6.2 Stress-strain behaviour of RHA based GPC

The stress strain behaviour of the RHA based geopolymer concrete using different curing conditions and ingredients have been shown with the help of the Figs. 5.8 to 5.10.



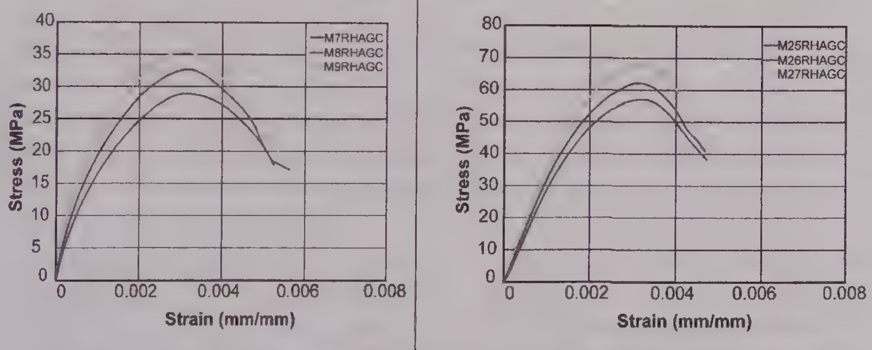
(a) Curing period at 27^0 C curing temperature ***(b) Curing period at 90^0 C curing temperature***

Fig. 5.8: Stress strain curves of GPC specimens using 8M NaOH



(a) Curing period at 27^0 C curing temperature *(b) Curing period at 90^0 C curing temperature*

Fig. 5.9: Stress strain curves of GPC specimens using 12M NaOH



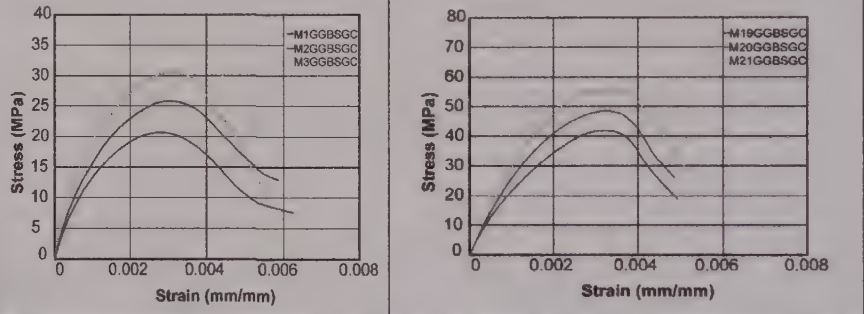
(a) Curing period at 27^0 C curing temperature *(b) Curing period at 90^0 C curing temperature*

Fig. 5.10: Stress strain curves of GPC specimens using 16M NaOH

The above curves have been drawn by testing the RHA based geopolymer concrete cylinders of size 150mm X 300mm, after 28 days. Initially, the trend of the line obtained was straight which shows the close relationship between the stress and strain. The stress strain curves show the typical mechanical response for the given loading and it is clear from the above figures that maximum compressive strength has been achieved at a strain value equals to in between 0.0022-0.0038, this is same as obtained by other researchers in the case of conventional concrete. This may be due to the nearly same value of the density of the GPC to conventional concrete. Further, ambiently cured RHA based GPC specimens failed with the large deformation instead of heat cured specimens.

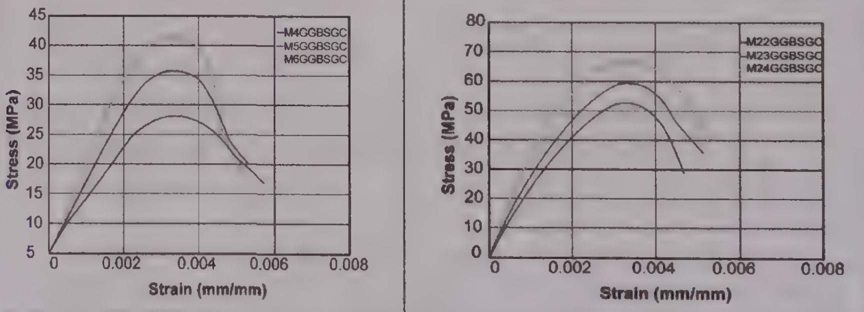
5.6.3 Stress-strain relationship of GGBS based GPC

Mixes M1GGBSGC to M27GGBSGC were tested to study the stress strain behaviour of the slag based geopolymer concrete.



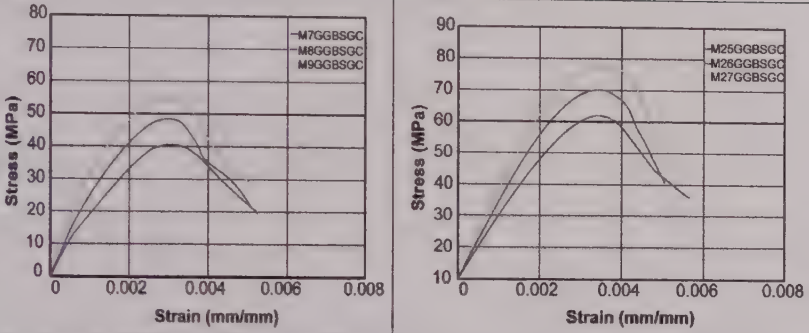
(a) Curing period at 27^0 C curing temperature *(b) Curing period at 90^0 C curing temperature*

Fig. 5.11: Stress strain curves of GPC specimens using 8M NaOH



(a) Curing period at 27^0 C curing temperature *(b) Curing period at 90^0 C curing temperature*

Fig. 5.12: Stress strain curves of GPC specimens using 12M NaOH



(a) Curing period at 27^0 C curing temperature

(b) Curing period at 90^0 C curing temperature

Fig. 5.13: Stress strain curves of GPC specimens using 16M NaOH

As discussed in the clause 5.5.1, all the cylindrical samples were tested under controlled conditions, load and strain readings were recorded till the time of failure. However, as the loading rate influence the strength, therefore the compressive strength of the test cylinders was low compare to that of cubes, as reported in Table 4.14. The stress strain behaviour of the geopolymer specimens with different NaOH molarity, GGBS content and curing temperature is shown in the above Figs. 5.11 to 5.13. It is worth noting from the above figures that GGBS based geopolymer behaves similar to conventional concrete, as maximum strength for the ambiently and heat cured specimens were achieved when the strain values were in between 2.5-3.8 x 10^{-3} mm/mm. Further, similar to FA based GPC, the rate of strain increase at the initial stage, was slower and the trend continued upto 80% of the maximum stress. The rate of increase in deformation of GGBS based geopolymer concrete was little bit faster than conventional concrete and this may be due to more amount of CaO in the matrix which further leads to large number of micro cracks near peak stress.

The obtained stress-strain curves were compared to the analytical model proposed by Junaid et al. [148] and the results are given in Fig. 5.14. As can be seen from the figure, the model proposed by Junaid et al. [148] was in good agreement with the experimental data of the selected specimen. It can therefore be concluded that the model may be used to predict the stress-strain behaviour of GPC samples under compression loading.

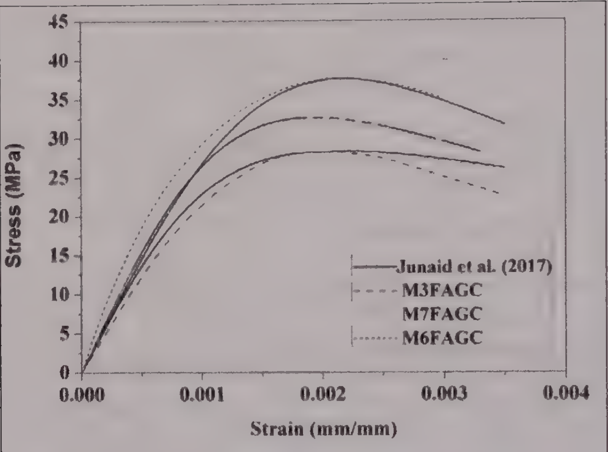


Fig. 5.14: Comparison of stress-strain data with analytical model.

From the above figures and discussions, it is ample clear that geopolymer concrete prepared with different alumino-silicate materials showed the similar range of strain at peak stress as observed by different researchers on conventional and fly ash based geopolymer concrete. Further, slope after peak stress in all the cases was on higher side which show the brittleness of the geopolymer concrete.

5.7 MODULUS OF ELASTICITY OF GPC

All the GPC mixes which were made to develop the design aids and to study the stress strain behaviour were further used to measure the modulus of elasticity. Also, these mix proportions covered compressive strengths ranging from 15MPa to 82MPa by utilizing the FA, RHA and GGBS as independent binder in addition to alccofine. The Young's modulus of geopolymer concrete was determined as secant modulus measured at the stress level equals to 33 percent of the average compressive strength obtained after testing the cylinders of standard size 150mm x 300mm and at the age of 28 days. Further, testing was done in accordance with Indian standards. Tables 5.8 to 5.10 shows the values of the Young's modulus of all the specimen's. As expected, the modulus of elasticity increased with the increase in binder's quantum, NaOH concentration and curing temperature, moreover, modulus of elasticity increased with the increase in compressive strength. Young's modulus for heat cured GPC specimens prepared with FA, RHA and GGBS along with alccofine ranges from 0.215 x 10^5

MPa to 0.381 x 10^5 MPa and 0.157 x 10^5 MPa to 0.313 x 10^5 MPa for ambient cured specimens.

American Concrete Institute (ACI) code and Indian Standard (IS) gives the elastic modulus as a direct function of the characteristics compressive strength for conventional concrete in terms of cylinders and cubes, which are shown in Eq. 5.4 and 5.5, respectively.

$$E_c = 4733 \times \sqrt{f_c} \quad (5.4)$$

$$E_c = 5000 \times \sqrt{f_c} \quad (5.5)$$

Where, E_c = *Elastic modulus and* f_c = *Compressive strength*

Table 5. 8: Modulus of elasticity or Young's modulus (E_m) of FA based GPC

Mixture	E_m, MPa ($\times 10^5$)	E_c, MPa ($\times 10^5$) As Per IS	E_m, MPa ($\times 10^5$)	E_c, MPa ($\times 10^5$) As Per ACI
M1FAGC	0.188	0.235	0.193	0.201
M2FAGC	0.205	0.251	0.197	0.212
M3FAGC	0.215	0.261	0.220	0.232
M4FAGC	0.227	0.274	0.231	0.246
M5FAGC	0.249	0.296	0.268	0.276
M6FAGC	0.259	0.309	0.259	0.288
M7FAGC	0.245	0.288	0.233	0.259
M8FAGC	0.266	0.309	0.254	0.276
M9FAGC	0.275	0.320	0.258	0.284
M19FAGC	0.237	0.297	0.266	0.280
M20FAGC	0.257	0.313	0.271	0.288
M21FAGC	0.274	0.332	0.294	0.303
M22FAGC	0.291	0.350	0.289	0.317
M23FAGC	0.303	0.361	0.298	0.331
M24FAGC	0.312	0.371	0.338	0.345
M25FAGC	0.343	0.403	0.366	0.382
M26FAGC	0.357	0.416	0.377	0.393
M27FAGC	0.367	0.427	0.384	0.404

Table 5. 9: Modulus of elasticity or Young's modulus (E_m) of RHA based GPC

Mixture	E_m, MPa (x 10^5)	E_c, MPa (x 10^5) As Per IS	E_m, MPa (x 10^5)	E_c, MPa (x 10^5) As Per ACI
M1RHAGC	0.179	0.224	0.182	0.189
M2RHAGC	0.192	0.240	0.192	0.206
M3RHAGC	0.201	0.251	0.206	0.217
M4RHAGC	0.221	0.266	0.218	0.232
M5RHAGC	0.242	0.288	0.247	0.255
M6RHAGC	0.253	0.301	0.241	0.268
M7RHAGC	0.239	0.281	0.221	0.246
M8RHAGC	0.258	0.300	0.246	0.268
M9RHAGC	0.269	0.312	0.255	0.280
M19RHAGC	0.230	0.288	0.242	0.255
M20RHAGC	0.253	0.305	0.256	0.272
M21RHAGC	0.268	0.324	0.283	0.292
M22RHAGC	0.282	0.343	0.282	0.310
M23RHAGC	0.294	0.354	0.289	0.321
M24RHAGC	0.306	0.364	0.325	0.331
M25RHAGC	0.337	0.397	0.349	0.364
M26RHAGC	0.348	0.409	0.361	0.376
M27RHAGC	0.358	0.421	0.368	0.387

Table 5. 10: Modulus of elasticity or Young's modulus (E_m) of GGBS based GPC

Mixture	E_m, MPa (x 10^5)	E_c, MPa (x 10^5) As Per IS	E_m, MPa (x 10^5)	E_c, MPa (x 10^5) As Per ACI
M1GGBSGC	0.179	0.221	0.179	0.186
M2GGBSGC	0.211	0.261	0.212	0.228
M3GGBSGC	0.237	0.289	0.244	0.257
M4GGBSGC	0.227	0.277	0.230	0.244
M5GGBSGC	0.263	0.309	0.269	0.277
M6GGBSGC	0.286	0.336	0.274	0.304
M7GGBSGC	0.275	0.323	0.262	0.291
M8GGBSGC	0.302	0.351	0.293	0.319
M9GGBSGC	0.325	0.377	0.314	0.345
M19GGBSGC	0.269	0.336	0.289	0.304
M20GGBSGC	0.302	0.364	0.311	0.331
M21GGBSGC	0.327	0.385	0.341	0.352
M22GGBSGC	0.308	0.375	0.312	0.343
M23GGBSGC	0.332	0.400	0.330	0.367
M24GGBSGC	0.354	0.421	0.380	0.387
M25GGBSGC	0.345	0.411	0.362	0.377
M26GGBSGC	0.368	0.433	0.383	0.399
M27GGBSGC	0.391	0.455	0.399	0.420

Therefore, equations derived for conventional concrete in ACI and IS overestimate the elastic modulus of the geopolymer concrete. Further, the reason for lesser values of the Young's modulus in the case of GPC could be the quality or stiffness of the aggregates, geometry of the matrix etc. Overall, geopolymer concrete reflects its ability to deform elastically similar to conventional concrete. Therefore, geopolymer concrete can be successfully implemented in compression structural elements such as columns, as the observed values of the elastic modulus can avoid the excessive deformation. Also, higher elastic modulus reduces the prestress loss in terms of elastic shortening thus, creates hope of its utilization in the prestressed geopolymer concrete industry while providing satisfactory serviceability.

5.8 RELATIONSHIP BETWEEN COMPRESSIVE AND SPLIT TENSILE STRENGTH

The relation between compressive and splitting tensile strength for alccofine activated GPC specimens is shown in Fig. 5.15. Previous researchers depicted a close relation between split tensile and compressive strengths of concrete and the same can be confirm in Table 5.11. Nonlinear equations proposed were based on regression analysis between tensile and compressive strength of concrete. The relationship between the compressive and tensile strength for alccofine activated geopolymer concrete is proposed by equation (5.6). It can be seen from the Fig. 5.15 that the regression line for geopolymer concrete can be regarded as a realistic representation, which can be applied to heat and ambient cured specimens, respectively. It has a direct relationship with the compressive strength similar to ordinary concrete.

$$f_{spt} = 0.46 \times f_c^{0.56} \text{ MPa} \qquad (5.6)$$

Where, f_{spt} = *Split tensile strength and* f_c = *Compressive strength in MPa*

Table 5. 11: Relationship between compressive and split tensile strength

	ACI 318-99 [164]	Gardner et al. [165]	Raphael et al. [166]	Carino et al. [167]	Current study	Ryu et al. [106]	Lee et al. [168]	Sofi et al. [48]	Anuradha et al. [169]
	Conventional concrete				Geopolymer concrete				
	Split tensile strength, $f_{spt} = \alpha * (\textit{Compressive strength}, f_c)^{\beta}$								
α	0.56	0.46	0.313	0.272	0.46	0.17	0.45	0.48	0.892
ß	0.50	0.60	0.66	0.71	0.56	0.75	0.50	0.50	0.422

Where, α and ß are constants.

5.9 RELATIONSHIP BETWEEN COMPRESSIVE AND FLEXURAL STRENGTH

Fig. 5.16 shows a scattered plot in between flexural and compressive strengths which were developed by using the relationships as shown in Table 5.12. The proposed equation is based on the nonlinear regression model and is shown in equation 5.7.

$$f_s = 0.66 \times \sqrt{f_c} \text{ MPa (5.7)}$$

Where, f_s = *Flexural strength and* f_c = *Compressive strength*

Table 5. 12: Relationship between compressive and flexural strength

	AS 3600 [170]	ACI 318 [164]	IS 456 [153]	NZS-3101 [171]	Current study	Gunasekara et al. 2017 [172]
	Conventional concrete				Geopolymer concrete	
	Flexural strength, $f_s = \alpha$ *sqrt (*Compressive strength,* f_c*)*					
α	0.60	0.62	0.70	0.60	0.66	0.70

Where, α and ß are constants.

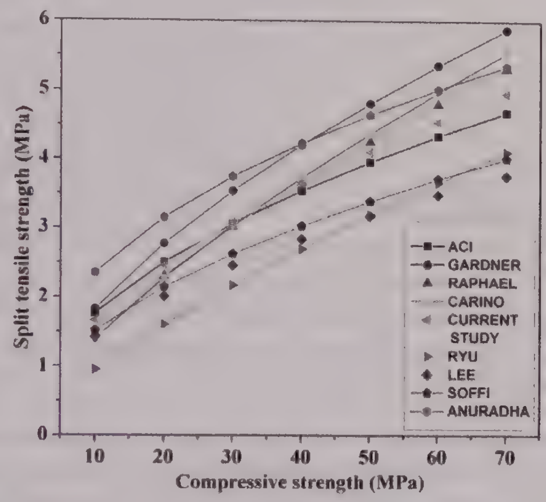


Fig. 5.15: Relationship between compressive and split tensile strength.

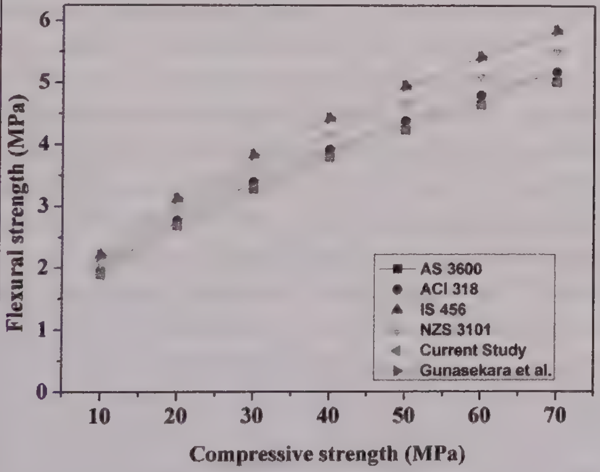


Fig. 5.16: Relationship between compressive and flexural strength.

It can be concluded from the Figs 5.15 and 5.16 that the nonlinear proposed equations for the geopolymer concrete fall within the already existing equations for conventional concrete in the various standards such as American Concrete Institute code (ACI), Australian Standard (AS) and Indian Standard (IS). In addition, the relationship developed by other researchers has also considered for comparison. It is clear from the above proposed equations that split tensile and flexural strength increases with the increase in compressive strength. It is worth noting that design equation provided by ACI and AS for conventional concrete underestimate the flexural strength. Further, IS overestimate the flexural strength but the difference noticed was much lower therefore, application of the ACI and AS would provide conservative design than IS in terms of flexural strength. Further, it was noticed from Fig. 5.14 that ACI design codes can be applied to geopolymer concrete in terms of split tensile strength. Overall, flexural strength and split tensile strength of the geopolymer concrete can be well predicted by using the proposed equations and can be utilised with confidence while designing the structural members.

Summary:- Alccofine (AF) together with the FA, RHA and GGBS have significant effects on the strength of the GPC. Geopolymer concrete follows similar trend to that of conventional concrete in terms of compressive, split tensile and flexural strengths, elastic modulus and stress-strain behaviour. Proposed rational mix design approach is suitable for designing the mix for geopolymer concrete cured at ambient as well as at elevated heat. The consistent results obtained in the validation procedure show the ability of the method to serve the industrial and commercial demands. In general, the results show that it is possible to target the workability and target compressive strength for geopolymer concrete which shows similar or better properties to conventional concrete. Maximum stress has been obtained at a strain value ranging from 0.0025 to 0.0035 and this was same for all the mixes. Further the strain at failure was in the range of 0.004 to 0.006, also modulus of elasticity ranges from 0.215 x 10^5 MPa to 0.381 x 10^5 MPa and 0.157 x 10^5 MPa to 0.313 x 10^5 MPa for heat and ambient cured specimens, respectively. These evidences are enough to show that GPC has similar properties to conventional concrete and structural design methodologies of the conventional concrete can be applied to GPC structural members.

CHAPTER - 6

CONCLUSIONS AND RECOMMENDATIONS

This chapter presents a set of conclusions of the research program, recommendations and scope for the future research on geopolymer concrete. Geopolymer concrete by incorporating alccofine along with FA, RHA and GGBS was developed and tested at different ages to study the structural properties. Apart from it, geopolymer concrete mix design procedure was developed and validated in the study.

6.1 CONCLUSIONS

Based upon the objectives, experimental programme undertaken and methodology adopted, the following conclusions can be derived upon:

1) Based on the present study, GPC prepared using un-processed fly ash does not gain any desirable structural properties, maximum compressive strength achieved was 14MPa even at 90^0C curing temperature and 16M NaOH solution.
2) The presence of alccofine in the geopolymer concrete containing processed fly ash led to the hydration process in addition to polymerization (as confirmed by XRD), this combined effect of hydration and polymerization (as confirmed by SEM) improved the fresh and hardened properties through the denser matrix, fewer microcracks and voids, at ambient and elevated temperatures.
3) Alccofine enhanced the workability of the geopolymer concrete in terms of slump, maximum value of slump was 160mm with 8M NaOH solution and 400kg/m^3 fly ash.
4) In this study, it is observed that inclusion of alccofine helped to achieve maximum compressive strength of 57MPa and 83MPa at 28 days by using 400kg/m^3 binder content, 16M NaOH concentration, W-GPB ratio of 0.27, ratios of silicate to hydroxide of 2.5 and AAL-GPB ratio of 0.45, at ambient (27^0C) and heat curing (90^0C), respectively.
5) In all the mixes with different concentration of sodium hydroxide, an increase in compressive, split tensile and flexural strengths were noticed with the increase in alumino-silicate materials quantum. It was observed that strength increased 15-18% and 11-14% when alumino-silicate material quantum changed from 350kg/m^3 to 375kg/m^3 and 375kg/m^3 to 400kg/m^3, further, the trend was same for all the alumino-silicate materials at ambient and elevated temperatures.

6) Increase in curing temperature has significant effects on strength and maximum compressive, split tensile and flexural strengths were observed at 90^0C for geopolymer concrete in all the cases.
7) A significant increase in the strength of the geopolymer concrete was noticed when concentration of the NaOH changed from 8M to 12M and 12M to 16M. In all the cases, the percentage increase in strength noticed was 18-21% and 15-18%, respectively.
8) High early age compressive strength at ambient and elevated temperatures could also be achieved by considering the GGBS as a binder into the matrix and incorporating alccofine (10%). With 400kg/m^3 GGBS and 16M NaOH concentration and at the age of 3 days, 45MPa and 70MPa of compressive strength could be achieved at ambient (27^0C) and elevated (90^0C) temperature, respectively.
9) The increase in early age strength when heat curing adopted was due to accelerated formation of calcium silicate hydrate (CSH) formed by the inclusion of alccofine in addition to the polymeric products CASH and NASH. Also, the calcium present in the alccofine reacted with the alkaline activators and produced heat which might have helped in increase rate of polymerisation.
10) Proposed mix design method which targeted the required compressive strength and workability of geopolymer concrete including FA, RHA and GGBS provided good results and was also validated.
11) The stress strain curves of the geopolymer concrete produced with different raw materials indicated brittle failure. Also, the strain at the peak stress were in between 0.0025 to 0.0038 as obtained for conventional concrete. Further, this was in the same range for FA, RHA and GGBS based GPC. Therefore, the behavior of GPC was similar to that of conventional concrete.
12) Young's modulus of elasticity of the geopolymer concrete was in the range of that has been specified in the Indian standards IS 456 valid for conventional concrete.
13) Structural design methodologies of the conventional concrete can be applied to GPC structural members.
14) Overall, this study demonstrates that geopolymer concrete has the same behaviour as obtained for conventional concrete but the mix design provisions of the conventional concrete could not be used to develop geopolymer concrete. Further, design aids have been developed for the different waste materials along with the steps to be followed while using them, to target the required workability and compressive strength.

6.2 RECOMMENDATIONS OF THE STUDY

1) By inclusion of alccofine along with waste raw materials (FA or RHA or GGBS), workable and high strength geopolymer concrete can be produced which can serve the cast in situ and precast concrete industries by following the proposed methodology of GPC mix design.
2) High early age strength of the GPC can be achieved particularly by incorporating alccofine in GPC based on GGBS at ambient temperature.
3) As the behaviour of the GPC was similar to conventional concrete, therefore, structural design methodologies of conventional concrete can be applied to geopolymer concrete.

6.3 SCOPE FOR FUTURE RESEARCH

From the results and conclusions reported from the analysis, following recommendations for future research are included:

1) The effect of alccofine on the geopolymer concrete have been examined only upto 90^0C curing temperature. Effects of elevated temperature on the newly developed geopolymer concrete in order to study fire resistance particularly in GPC based on GGBS is necessary.
2) Durability aspects of the fly ash, rice husk ash and GGBS based geopolymer concrete with the inclusion of the alccofine can be explored.
3) This study reports the inclusion of the alccofine to achieve the required slump and compressive strength at different temperatures with NaOH and Na_2SiO_3 as an alkaline activator. However, the properties of the concrete can be examined by using different alkaline activators such potassium hydroxide, sodium carbonate etc.
4) Fly ash, RHA and GGBS from different sources in India has different chemical and physical properties. Systematic approach is required to cover the variation in the properties and exploit the potential of these waste materials for better sustainability.

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LIST OF PUBLICATIONS

Sr. No	Title/Journal Details	Remarks
1	**Mechanical and microstructural properties of fly ash based geopolymer concrete incorporating alccofine at ambient temperature.** *Construction and Building Materials 2017.*	Accepted
2	**Predicting relationship between mechanical properties of low calcium fly ash based geopolymer concrete.** *Transactions of the Indian ceramic society 2017.*	Accepted
3	**Development of mix design method for geopolymer concrete.** *Advances In Concrete Construction* 2017, **5:** 377-390.	Published
4	**Experimental study on geopolymer concrete prepared using high-silica RHA incorporating alccofine.** *Advances In Concrete Construction* 2017, **5:** 345-358.	Published
5	**Improving compressive strength of low calcium fly ash geopolymer concrete with alccofine.** *Advances In Concrete Construction* 2017, **5:**17-29.	Published
6	**Preparation of Geopolymer Concrete (GPC) Using High-Silica Rice Husk Ash (RHA) Incorporating Alccofine.** *Advanced Science, Engineering and Medicine* 2017, **9:**370-376.	Published
7	**Suitability of Ambient-Cured Alccofine added Low-Calcium Fly Ash-based Geopolymer Concrete.** *Indian Journal of Science and Technology* 2017, **10**.	Published
8	**Prediction of Mechanical Properties of Alccofine Activated Low Calcium Fly Ash Based Geopolymer Concrete.** *ARPN Journal of Engineering and Applied Sciences* 2017, **12:**3022-3031.	Published
9	**Mechanical Properties of GGBS Based Geopolymer Concrete Incorporating Alccofine with different Concentration and Curing Temperature.** *Advanced Science, Engineering and Medicine* 2017	Published
10	**Behaviour of fly ash based geopolymer concrete in fresh state.** *Indian Journal of Science and Technology* 2017, 10.	Published
	Conference paper title/ Detail	
1	**Mechanical properties of geopolymer concrete - a state of the art report.** *5th Asia And Pacific Young Researchers And Graduate Symposium-13 (MNIT Jaipur).*	

PATENT

1 “Composition of high compressive strength low calcium fly ash based geopolymer concrete incorporating Alccofine 1203” CBR No. 14421, Application No. 201711012671 filed with IPR India dated 07-04-2017.

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Geopolymer concrete has emerged as a novel construction material and is one of those technologies where research is attempting for the last two decades to replace with OPC. Also, it has been established that geopolymer can act as an alternative binder to OPC in the concrete. Apart from its several technical advantages like good physical, chemical and mechanical properties geopolymer concrete adds sustainability to the environment as it has significantly smaller greenhouse footprint than Portland cement binders. Geopolymer binders are a class of aluminosilicates based materials. Geopolymers are synthesized by thermal activation of solid aluminosilicate materials such as fly ash (FA), metakaolin, rice husk ash (RHA), granulated blast furnace slag (GGBS), etc., with an alkali metal hydroxide and silicate solution.

Dr Parveen is developing new structures which are greener, cheaper, and longer lasting. He is targeting improving the prefab construction technology with tremendous time and cost savings. In addition, he is developing rubberised concrete which turns waste car tyres toward possible infrastructural applications.

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