PERFORMANCE EVALUATION OF GEOPOLYMER MORTAR MIXES USING RECYCLED FINE AGGREGATE

Thesis

submitted in partial fulfilment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

by

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DECLARATION

I hereby *declare* that the Research Thesis entitled **PERFORMANCE EVALUATION OF GEOPOLYMER MORTAR MIXES USING RECYCLED FINE AGGREGATE** which is being submitted to **National Institute of Technology Karnataka, Surathkal,** for the partial fulfilment of the requirement for the award of degree of **Doctor of Philosophy** in the **Department of Civil Engineering**, is a *bonafide report of the work carried out by me*. The material contained in this Research Thesis has not been submitted to any university or Institution for the award of any degree.

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Date: 02-03-2019

CERTIFICATE

This is to *certify* that the Research Thesis entitled **PERFORMANCE EVALUATION OF GEOPOLYMER MORTAR MIXES USING RECYCLED FINE AGGREGATE** submitted by Mr. **SUMAN SAHA (Register Number: 155006CV15F12),** as the record of the research work carried out by him, is *accepted as the Research Thesis submission* in partial fulfilment of the requirements for the award of degree of Doctor of Philosophy.

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ABSTRACT

Manufacturing of Ordinary Portland cement (OPC) requires huge quantity of natural resources, energy and it releases large amount of carbon-dioxide to the environment. Numerous research efforts have been made continuously to establish geopolymer as the most suitable alternative binder material in view of economical and environment consideration. With the rapid growth in construction activities, high amount of construction and demolition waste (C&DW) is generated and large volumes of natural resources are also being consumed by the construction industry. As a result, both the quantities of C&DW to be disposed off and the scarcity of natural resources are increasing day by day. To promote sustainability in construction industry, the use of recycled concrete aggregates, resourced from C&DW is very important and provides a useful solution for the production of concrete. Here an attempt is made to use C&DW as recycled fine aggregate (RFA) for the production of fly ash based geopolymer mortar mixes. Effects of the concentration of sodium hydroxide solution, the ratio of sodium silicate solution to sodium hydroxide solution, the ratio of alkaline liquid to binder, different curing regimes and RFA content on the properties of mortar mixes produced have been explained based on the observations at laboratory. Experimental results indicate that 20% RFA can be used effectively to obtain better performances of the mixes. Using the observed results in the laboratories, prediction models for water absorption capacity, compressive strength and drying shrinkage of the produced geopolymer mortar mixes were developed using artificial neural networks.

Keywords: Construction and demolition waste, recycled fine aggregates, sustainability, mortar, fly ash, geopolymer, compressive strength, drying shrinkage.

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ABBREVIATIONS

OPC	:	Ordinary Portland Cement
C&DW	:	Construction and Demolition Waste
CO_2	:	Carbon - dioxide
FA	:	Fly Ash
GGBFS	:	Ground Granulated Blast Furnace Slag
MT	:	Million Tonnes
FY	:	For the Year
CAGR	:	Compound Annual Growth Rate
C-S-H	:	Calcium Silicate Hydrate
Si	:	Silicon
Al	:	Aluminium
SH	:	Sodium Hydroxide
SS	:	Sodium Silicate
AL	:	Alkaline Liquid
SS/SH	:	Ratio of sodium silicate solution to sodium hydroxide solution
AL/FA	:	Ratio of alkaline liquid to fly ash
NFA	:	Natural Fine Aggregate
RFA	:	Recycled Fine Aggregate
IS	:	Indian Standard
ANN	:	Artificial Neural Network

CHAPTER 1

INTRODUCTION

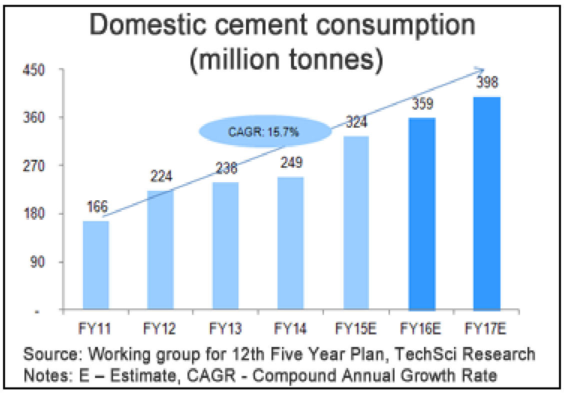
1.1 GENERAL

Construction industry is growing rapidly day by day. In the construction industry, concrete is used as the premier construction material across the world and the most commonly used in all types of civil engineering works including infrastructure, building works, defence installations etc. Ordinary Portland Cement (OPC) is the main binder material in concrete. As the demand for concrete as construction material increases, the demand for OPC is also increased. Production of OPC requires huge quantity of natural resources and also releases a huge quantity of carbon-dioxide (CO_2) into the atmosphere due to the calcination of limestone and the combustion of fossil fuels during the process. As sustainable development and environmental protection become very important to all industries in recent years, research efforts are continuing throughout the world to make construction industry more sustainable and eco-friendlier by recycling the materials, reducing the use of OPC and by using waste industrial by-products such as fly ash, ground granulated blast furnace slag, silica fume etc. as partial or full replacement of OPC.

On the other side, regarding construction materials, the main problems are a huge amount of construction and demolition waste (C&DW) generated by the construction industry and landfill space depletion also. Depletion of natural resources becomes a very important matter to look into. So, recycling of the waste materials has gained greater momentum to promote sustainability in the industries. Recycled concrete aggregates are the main components of old concrete and for many reasons, there is a need to re-use them. As because there is going to be a critical shortage of natural virgin aggregates for production of fresh concrete, and the enormous amounts of demolished concrete produced from deteriorated and obsolete structures create severe ecological and environmental problems. Recycling of aggregate materials from C&DW may reduce the demand-supply gap effectively.

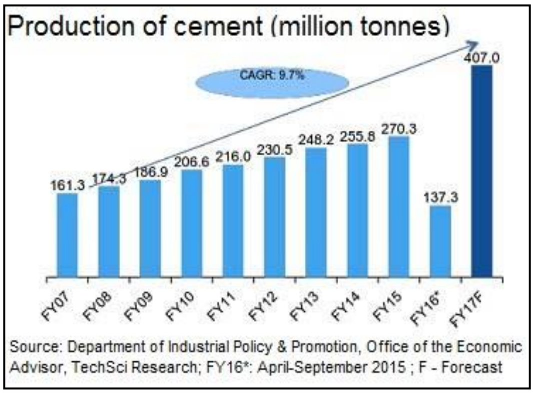
1.2 DOMESTIC CEMENT CONSUMPTION IN INDIA

With nearly 390 million tonnes (MT) of cement production capacity, India is the second largest producer of cement in the world. Production of cement in India accounts for 6.7% of the world's cement output. The cement production capacity is estimated to touch 550 MT by FY - 2020. India's cement production increased at a compound annual growth rate (CAGR) of 6.7% to 270.32 MT over FY-2007 – FY-2015. As per the Twelfth Five Year Plan, production is expected to reach 407MT by FY-2017. The consumption is further expected to increase at a CAGR of 15.7% during FY2011- FY2017. Demand is increasing due to the infrastructure development in tier-2 and tier-3 cities. The country's per capita consumption of cement is around 190 kg, which is lower than the World average, which is around 500 kg. This provides further scope for the demand in the industry. Figure 1.1 and Figure 1.2 show the scenario of the consumption and production of cement in the country respectively.



Source: Working group for 12th Five Year Plan, TechSci Research
Notes: E – Estimate, CAGR - Compound Annual Growth Rate

Figure 1.1 Domestic Cement Consumption



Source: Department of Industrial Policy & Promotion, Office of the Economic Advisor, TechSci Research; FY16*: April-September 2015 ; F - Forecast

Figure 1.2 Production of Cement

Source : http://www.ibef.org/industry/cement-india.aspx (15-01-2016 @ 19:30 hrs)

1.3 USE OF FLY ASH

The availability of fly ash throughout the world creates huge opportunities to utilize this by-product material of burning coal, as partial replacement or as performance improver for OPC. Partial replacement of OPC in concrete has become a more common practice that offers benefits to both the properties of the concrete and the environment. As partial replacement for OPC, fly ash reduces the need for OPC in concrete mixtures, contributing to the reduction of CO_2 emissions, and makes use of one waste by-product material, in which the binding properties do not exist. When fly ash is added to OPC as partial replacement or as an admixture, fly ash reacts with the calcium hydroxide released during the hydration process of OPC to form the calcium silicate hydrate (C-S-H) gel. The development and application of high volume fly ash concrete, which enabled the replacement of OPC up to 60% - 65% by mass (Malhotra, 2002; Malhotra and Mehta, 2002), can be considered as a landmark in this attempt. In the financial year 2016-17, the production of fly-ash is expected to increase around 300 - 400 MT/year (Md Emamul Haque, 2013). If this large amount of fly-ash is not utilized effectively in increased quantity, then it will be hazardous to the environmental systems.

1.4 CONSTRUCTION AND DEMOLITION WASTE

Construction and demolition waste (C&DW) is defined as the solid waste generated during the construction, remodeling, renovation, repair, alteration or demolition of residential, commercial, government or institutional buildings, industrial, commercial facilities and infrastructures such as roads, bridges, dams, tunnels, railways, and airports etc. C&DW constitutes a major portion of total solid waste production in the world. It consists of different materials such as concrete, metal, wood, plastics, bricks etc. In addition, it includes the materials generated as a result of natural disasters. C&DW can be classified into two components; major components include cement concrete, bricks, cement mortar, steel from reinforced cement concrete, doors & windows, roofing support systems, rubble, stones, timber etc. and minor components includes conduits, galvanized iron pipes/iron pipes/plastic pipes, electrical fixtures, panels, glass etc. The quantity of C&DW is increasing day by day due to the rapid growth in the construction industry. The main reasons for the increase of the volume of C&DW are as follows:

- Many old buildings and structures have overcome their limit of use and need to be demolished.
- Structures even adequate to use, are under demolition because there are new requirements and necessities.

- Creation of building wastes which result from natural destructive phenomena (earthquakes, storms. etc).
- New construction for better economic growth.
- Creation of building waste resulting from manmade disaster/war.

1.5 NEED FOR THE STUDY

Manufacturing of Portland cement is a resource exhausting, energy-intensive process that releases large amounts of the greenhouse gas CO_2 into the atmosphere. Production of 1 tonne of Portland cement requires about 2.8 tonnes of raw materials, including fuel and other materials. As a result of de-carbonation of lime, the manufacturing of 1 tonne of cement generates about 1 tonne of greenhouse gas. Neville (1997) stated that the cement manufacturing industries release approximately 1 tonne of carbon-dioxide into the atmosphere to produce 1 tonne of OPC. This is approximately 5% to 7% CO_2 emissions of the global CO_2 emissions. As production of OPC consumes hugequantity of raw materials and releases a high amount of carbon - dioxide into the atmosphere, there is an immense need to develop alternative materials to protect the natural resources and environment.

After the invention of geopolymer by Prof. Joseph Davidovits in the 1970s, lot of research has been carried out to find the suitability to use geopolymer as an alternative binder material. Previous investigations have shown that curing at a certain range of temperature (heat cured) can enhance the strength development of geopolymer. To use geopolymer in practical applications, it is not possible always to provide heat curing. In the present study, efforts are made to develop ambient cured geopolymer mortar mixes for practical applications in the construction industry. Additionally, investigation on heat cured geopolymer mortar mixes also has been done for comparing their performance with that of ambient cured geopolymer mortar mixes.

On the other hand, the concrete industry consumes large quantities of natural resources worldwide, which are becoming insufficient to meet increasing demands. Consumption of natural resources in large quantity also leads to the damage to the environment. Due to excessive cost in extraction and transportation of natural resources like coarse aggregates, and fine aggregates are becoming expensive in the industry. As the construction industry is growing day by day, the demand of these natural resources is also in increasing trend. As a result, both the environmental problems and scarcity of natural resources co-exist in present scenario. Therefore, there is a necessity to find alternative materials for aggregates also. In addition, quantity of C&DW is also increasing rapidly due to huge infrastructural development in the present scenario. Unscientific, unplanned dumping of this C&DW results in lot of problems to the environment. So, it will be helpful to find effective ways to use these waste materials efficiently for the protection of the environment. In this present study, an effort has been made to use C&DW effectively as recycled fine aggregate for the alternative material of natural fine aggregate (river sand).Therefore present research work aims the following:

- To use fly ash based geopolymer effectively as alternative eco-friendly binder material for OPC.
- Reuse the C&DW as recycled aggregate effectively.
- Promoting sustainability in the construction industry.

1.6 ORGANIZATION OF THESIS

This thesis is structured in the following format.

Chapter 1: Introduction: It presents various aspects of cement consumption, usage of fly ash, construction and demolition waste, recycled aggregates. Based on this basis, the need for the present investigation has been identified.

Chapter 2: Review of Literature: A review of recent literature on geopolymer mixes, alternative materials to be used as aggregates has been discussed. This chapter also explainstheobjectives and scope adopted for this investigation.

Chapter 3: Materials and Methodology: The properties of all the materials used to cast the test specimens, the sizes and the number of specimens, testing procedures and the associated instrumentations are described in details in this chapter.

Chapter 4: Results and Discussion: All the observations from the different tests on the cast specimens, analysis of the results, and the related discussion have been included in a sequential way. The results and discussion of this experimental work have been presented briefly.

Chapter 5: Prediction Model using ANN: All the properties observed from this experimental study were used to create prediction models using the artificial neural network. The ANN model to predict the properties have been discussed briefly.

Chapter 6: Conclusion: The important conclusions, based on the observations made in the experimental investigations, have been integrated and presented in a logical sequence and recommendations are made.

CHAPTER 2

REVIEW OF LITERATURE

2.1 GENERAL

In this chapter, a review of the literature in the field of geopolymer mortar and concrete mixes has been carried out in details. A brief review of the published works on the materials used for the production of geopolymer mortar/concrete mixes and the properties of geopolymer mortar/concrete mixes is presented. Finally, the objectives of the present investigation are identified.

2.2 GEOPOLYMERTECHNOLOGY

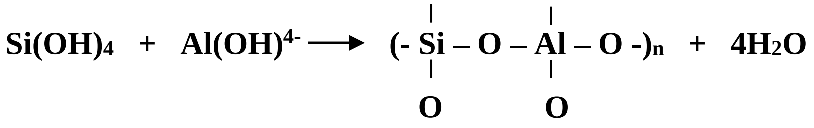
The term ‘geopolymer’ was invented in the 1970s by the French scientist and engineer Prof. Joseph Davidovits, and is applied to a class of solid materials synthesized by the reaction of an alumina-silicate powder with an alkaline solution (Davidovits, 1991). Geopolymers are a member of the family of inorganic polymers and will have a chain structure formed on the support of Al and Si ions mainly. The chemical composition of this geopolymer material is similar to natural zeolitic materials, but they have amorphous microstructure instead of crystalline (Palomo et al., 1999; Xu and van Deventer, 2000). The polymerization process involves a substantially fast chemical reaction on Si-Al minerals under the highly alkaline condition, which results in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds, as follows (Davidovits, 1999):

$$M_n[-(SiO_2)\ z\text{–}AlO_2]_n.\ wH_2O$$

Where: M = the alkaline element or cation such as potassium, sodium or calcium; the symbol “–” indicates the presence of a bond, n is the degree of polycondensation or polymerization; z is 1, 2, 3, or higher, up to 32.

The schematic formation of geopolymer material can be shown as described by equation (2.1) and equation (2.2) (Davidovits, 1999). These formations indicate that all materials containing mostly Silicon (Si) and Aluminium (Al) can be processed to make the geopolymer material.

$$(Si_2O_5.Al_2O_2)_n + H_2O + OH^{-} \longrightarrow Si(OH)_4 + Al(OH)^{4-}$$ …(2.1)



$$Si(OH)_4 + Al(OH)^{4-} \longrightarrow (-\underset{\underset{O}{|}}{\overset{|}{Si}} - O - \underset{\underset{O}{|}}{\overset{|}{Al}} - O-)_n + 4H_2O$$ …(2.2)

A geopolymer can take one of the three basic forms (Davidovits ,1999, 2002), i.e. Poly(sialate), which has [-Si-O-Al-O-], Poly(sialate-siloxo), which has [-Si-O-Al-O-Si-O-] and Poly(sialate-disiloxo), which has [-Si-O-Al-O-Si-O-Si-O-]. “Sialate” denotes silicon-oxo-aluminate and it is used as an abbreviation. The molecular structure of those sialate forms is shown in Figure 2.1.

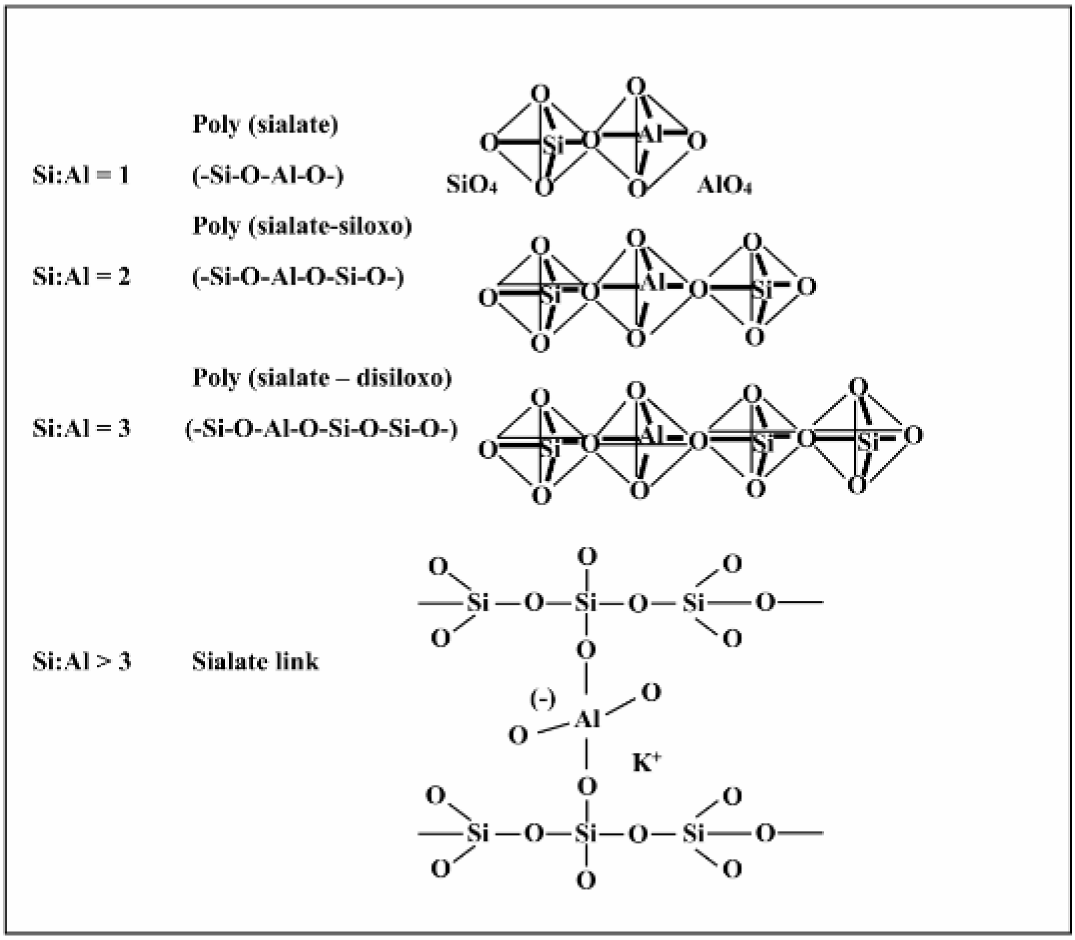


Figure 2.1 Molecular Structure of Geopolymer

Duxson et al. (2007) have given a brief description of the conceptual model of geopolymerization as shown in Figure 2.2.

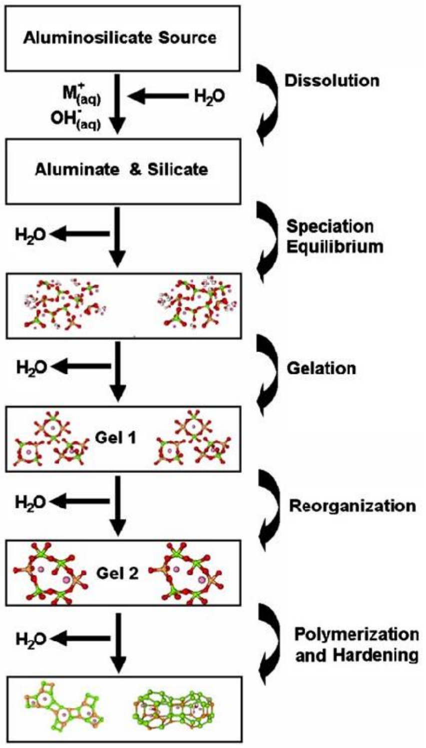


Figure 2.2 Model of geopolymerization (Duxson et al., 2007)

2.3 FACTORS AFFECTING THE PROPERTIES OF GEOPOLYMER MORTAR MIXES

For twenty-five years, geopolymer science was studied in very few laboratories and institutions. But during the last ten years, research on geopolymer has increased extensively throughout the world. The challenges of using geopolymer, the methods to be adopted during mixing, the factors to be considered etc. noted from a brief review of previous studies on geopolymer mortar and concrete mixes is presented here. From the previous research work, it can be said that the factors, which play important role on the properties of geopolymer mortar mixes, are concentration of Sodium Hydroxide (SH) solution, ratio of Sodium Silicate (SS) solution to the Sodium Hydroxide (SH) solution, modulus of alkali activator, curing conditions, and alkali liquid to fly ash/slag ratio etc. The effect of these different factors on the properties of geopolymer mixes is going to be reviewed from the past studies.

2.3.1 Effect of Alkaline Solution

Alkaline solutions such as sodium/potassium hydroxide (NaOH/KOH) solution and soluble silicates have a significant role on the properties of geopolymer mortar or concrete mixes. The effects of the addition of chemical activators on the physical, mechanical, durability, shrinkage, and microstructuralproperties of geopolymers are deliberated in detail as follows.

Neto et al. (2008) studied drying shrinkage and autogenous shrinkage of mortar samples prepared with activated slag cement. Samples were prepared with the consideration of the Na_2O dosage of 2.5%, 3.5% and 4.5% by the mass of slag. Sodium silicate having silica modulus 1.7 was used in this study. Considerable increment of drying and autogenous shrinkage and early hydration were observed with the increase of silica in activator solution. They concluded that extensive water loss when the samples were exposed to the environment and less water loss due to the hydration process might be the reasons for drying shrinkage. Refinement of activated slag cement of porous system was marked as the reason for autogenous shrinkage.

Thockhom et al. (2009) studied the resistance of geopolymer mortar samples produced with different percentage level of Na_2O in alkali activator solution kept in 10% sulphuric acid solution for a period of 18 weeks. Na_2O content varied from 5% to 8 % of fly ash. The ratio of water to binder and ratio of sand to fly ash were kept as 0.33 and 1.0 respectively. Cast specimens were kept at 85°C for 48 hrs for heat curing. Based on their study, it is concluded that geopolymer specimens lost its alkalinity within 15 weeks in the above-mentioned acid solution. After keeping samples in acid solution, weight loss of the geopolymer samples showed less than OPC samples.

Guo et al. (2010) carried out a study on compressive strength and microstructural characteristics of class C type of fly ash based geopolymer. The investigation was carried out at modulus of the mixed alkali activator 1.0, 1.5, 2.0 and 5% - 15% mixed alkali activator content by the mass of Na_2O to class C fly ash. The ratio of water to the class C fly ash was kept at 0.4 (by mass). The optimum modulus of mixed alkali activator was found to be 1.5 and content of alkali activator (by mass of Na_2O to fly ash) be 10% to get high compressive strength. From the microstructural analysis, they concluded that calcium silicate hydrate was formed along with geopolymeric gel i.e. sodium-poly (sialate-disiloxo) and some un-reacted spheres also observed.

Vargas et al. (2011) investigated the effects of the molar ratio (Na_2O/SiO_2) on the properties of alkali-activated fly ash geopolymer materials.97% sodium hydroxide (NaOH) was used as the alkaline activator in this study. The molar ratio was varied at 0.20, 0.30 and 0.40.High compression strength was observed for the mortar samples prepared with the alkali solution of molar ratio 0.40 and denser morphology was noticed for the paste samples of geopolymer prepared with the alkali solution of molar ratio 0.40.

Ridtirud et al. (2011) studied the effects of the concentration of sodium hydroxide (NaOH) solution and the ratio of sodium silicate (Na_2SiO_3) to sodium hydroxide solution on the shrinkage of fly ash based geopolymers. In this experiment, the ratio of sodium silicate to NaOH and the ratio of liquid to ash were used as 0.67 and 0.6 respectively. For this test, 40 °C temperature was used as curing temperature of samples. Concentrations of NaOH Solution were taken as 7.5M, 10.0M, and 12.5M. Based on results, they concluded that the concentration of NaOH solution has little effect on compressive strength, but significant effects on shrinkage. High shrinkage was observed for geopolymer produced with 12.5M NaOH solution comparing to that with a lower NaOH concentration. In another phase, different ratios of sodium silicate to sodium hydroxide solution (0.33, 0.67, 1.0, 1.5, and 3.0) were taken to prepare the samples of geopolymer mortars for testing. Low drying shrinkage of geopolymers was observed for high sodium silicate to NaOH ratio of 3.0 as compared to other geopolymers produced with the other ratios of sodium silicate to NaOH (0.3 - 1.5). In another phase of this research work, the ratio of liquid to ash was varied as 0.4, 0.5, 0.6, and 0.7 for the preparation of geopolymer samples. Significant increase in shrinkage was found with the increase in the ratio of liquid to ash from 0.4 to 0.7.

Somna et al. (2011) studied the compressive strength of Ground Fly Ash (GFA) geopolymer cured at ambient temperature by varying the NaOH concentration from 4.5M to 16.5M. Significant increase in the compressive strength of paste samples was observed with the increase of NaOH concentrations from 4.5M to 9.5M. When the concentration of NaOH solutions varies from 9.5M to 14M, then increase in the compressive strength of the paste sample was observed, but in a much lesser extent. This study suggested the higher degree of silica and alumina leaching as the reason fortheincrease in compressive strength with the increase of NaOH concentrations. At the NaOH concentrations of 16.5M, the compressive strength of GFA geopolymer pastes started to decline. Due to the excess hydroxide ions, the precipitation of alumina-silicate gel occurred at very early ages, thus results in lower strength of geopolymer pastes.

Dutta et al. (2012) did a comparative study on the properties of geopolymer paste prepared with different alkali activators. Potassium hydroxide (KOH) and sodium hydroxide (NaOH) were used as alkali activators. Sodium silicate solution was added to hydroxide solution 3 hours before being used to prepare geopolymer specimens and SiO_2/Na_2O or SiO_2/K_2O ratio was maintained at 1 and 1.5. Cast specimens were kept at 85°C for 48 hours for curing. Experimental results show higher compressive strength for specimens activated by potassium hydroxide solution when the ratio SiO_2/K_2O is 1.5 and higher compressive strength for specimens activated by sodium hydroxide solution when the ratio SiO_2/Na_2O is 1. Samples activated by sodium hydroxide solution exhibited little lower early strength than the strength after 28 days for specimen prepared with potassium hydroxide solution. Significant increase in compressive strength was observed with time for specimens activated by sodium hydroxide solution.

Chindaprasirt et al. (2012) investigated the influence of silica and alumina contents on physical properties of high calcium fly ash geopolymers. By adding nano-silica, RHA, or nano-alumina to mixes, the contents of silica and alumina varied whereas the control mix prepared with fly ash, sodium silicate, and sodium hydroxide solution in required quantities. In this experimental work, the contents of fly ash and water were kept constant in all mixes. Acceleration of setting of geopolymer was observed with the increase of alumina and silica both. The optimal SiO_2/Al_2O_3 ratio was found to be in the range of 3.20 - 3.70. Reduction in strength was also noticed while increasing the silica content ($SiO_2/Al_2O_3 > 4.3$) for high calcium fly ash based geopolymers.

Nagan et al. (2013) conducted an experiment to find the effects of the concentration of NaOH, ratio of sodium silicate to NaOH by mass on the workability, compressive strength and fire resistance of geopolymer mortar mixes. They concluded that the ratio of sodium silicate to NaOH and the concentration of NaOH have significant influences on the workability of the mortar mixes. But, with the exposure of 900 ºC, the strength of mortar was found to be decreased and found pH value to be high for the protection of the passive layer of steel.

Sukmak et al. (2013) studied the effect of the ratio of sodium silicate to sodium hydroxide (Na_2SiO_3/NaOH) and the ratio of liquid alkaline activator to Fly Ash (L/FA) on the development of compressive strength of clay-FA geopolymer bricks. In this study, Na_2SiO_3/NaOH ratios were taken as 0.4, 0.7, 1.0, 1.5 and 2.3 and the L/FA ratios were taken as 0.4, 0.5, 0.6 and 0.7 by dry clay mass. Tests were performed on the 7, 14, 28, 60 and 90 days of curing ages to find out the compressive strength of the cast brick samples. From the results, the optimum values of Na_2SiO_3/NaOH and L/FA ratios were found to be 0.7 and 0.6 respectively. The optimum Na_2SiO_3/NaOH ratio of0.7 is less than that of FA-based geopolymers as the clay had high cation absorption capability and might have absorbed some of the NaOH added. Clay-FA geopolymer bricks exhibited a significant decrease in strength when excessive alkali activator (L/FA > 0.6) was adopted due to the precipitation of dissolved Si and Al species at the early stages before the starting of the polycondensation process, resulting in cracks over the FA particles.

Budh and Warhade (2014) studied the effect of the concentration of sodium hydroxide solution on compressive strength of geopolymer mortar keeping constant the ratio of alkaline liquid to fly ash as 0.5, water to binder ratio as 0.30 and sand to fly ash ratio 1.0. The ratio of sodium silicate to sodium hydroxide is maintained as 1.0. The concentration of sodium hydroxide solution varied from 8M to 14M. Results showed high compressive strength and high Ultra Sonic Pulse Velocity for the higher concentration of the alkaline solution.

Gorhan and Kurklu (2014) investigated the influence of the sodium hydroxide solution on the properties of the class-F type of fly ash-based geopolymer mortar, which were cured at different temperatures and curing time. Three different concentration level of sodium hydroxide (NaOH) solution (3M, 6M,and 9M) were used with sodium silicate solution. It was found that concentration of NaOH solution had an effect on the properties of the mortar cured at different temperatures. Compressive strength values of the geopolymer mortars cured at 85°C increased depending on the curing time and the increase in the concentration of NaOH solution. They concluded that the optimal concentration of NaOH solution was 6M.

Kotwal et al. (2015) executed an experimental program to determine a relationship between the activator composition and the properties of geopolymer mortar based on class C fly ash in fresh and hardened states. In this program, scanning electron microscopy, energy dispersive X-ray spectroscopy and X-ray diffraction techniques were also used to characterize the geopolymer material. Temperature of fresh geopolymer mortar was in the range of from 32°C to 54°C. They observed increment of the temperature of mortar with the higher levels of NaOH and Na_2SiO_3. They found no significant variation in density due to specimen age or different alkali activator components. Flow was observed to be in decreasing trend while increasing NaOH component and fine aggregate content. The optimum ratio of NaOH to fly ash and the ratio of Na_2SiO_3 to fly ash were found to be 0.10. Based on their experiment program, they concluded that fly ash based geopolymer can be an alternative to Portland cement for the concrete industry.

Singh et al. (2016) conducted an experiment to find out the influences of the concentration of alkaline activator on the compressive strength, ITZ and drying shrinkage of flyash/slag based geopolymer concrete. They varied the concentration of activators from 10M to 16M and considered the ratio of sodium silicate to sodium hydroxide solution is 2.5. Fly ash and GGBS were mixed in the ratio of 2:1. Optimum activator concentration was found to be 14M for showing highest compressive strength due to hard microstructure. After 180 days, 1045 micro-strain (approximately 0.1%) only was observed as drying shrinkage of the geopolymer samples and also found that approximately 89% of the total shrinkage was obtained after 28 days.

2.3.2 Curing Conditions

Guo et al. (2010) studied the effects of curing temperature on the compressive strength of class C type of fly ash based geopolymer. Curing temperature of the sample was varied from 60°C to 90°C for the duration of 4 hrs, 8 hrs and 24 hrs followed by curing at 23°C temp for 28 days. From the test results, curing at 75°C for 8 hrs followed by curing at 23°C for 28 days was observed the optimum curing condition to obtain the highest compressive strength of the samples.

Somna et al. (2011) concluded from their study that sodium hydroxide activated ground fly ash geopolymer paste can be produced with reasonable strength at room temperature. NaOH concentrations are mentioned as the attribute for the variation of the compressive strength and microstructure of the geopolymer pastes.

Chindaprasirt et al. (2011) studied the effect of fineness of high calcium fly ash on high strength geopolymer paste and mortar. 10 M sodium hydroxide solution (NaOH) and the ratio of sodium silicate solution (Na_2SiO_3) to sodium hydroxide solution as 1.0 were kept throughout the experiment. Water (including water in NaOH solution and Na_2SiO_3) to solid mass ratio was kept also constant as 0.2. 0 hr, 1 hr, 2 hrs, 3 hrs, 6 hrs, and 24 hrs of delay time was provided before keeping for heat curing at the curing temperature of 75 °C for 48 hrs. After finding optimum delay time (1 hr), different curing temperatures of 30°C, 45°C, 60°C, 75°C, and 90°C also were used and duration in the oven was 48 hrs. After finding optimum delay time (1 hr) and curing temperature (75°C), duration of heat curing was used as 1, 2, 3, 4, and 5 days. Based on results, the optimum parameters for getting best properties (setting time, workability, high strength etc.) were found to be 1 hr of delay time and 3 days of heat curing at 75°C for fine fly ash activated with Na_2SiO_3 solution and 10M NaOH solution at the ratio of 1.0.

Ridtirud et al. (2011) studied the effects of different curing temperatures on the shrinkage of fly ash based geopolymers. In this experiment, the ratio of sodium silicate to NaOH and the ratio of liquid to ash were used as 0.67 and 0.6 respectively to prepare the geopolymer mortar samples. Concentration of NaOH solution was taken as 10M. The curing at 25, 40, and 60°C for 24 h was adopted for this test.

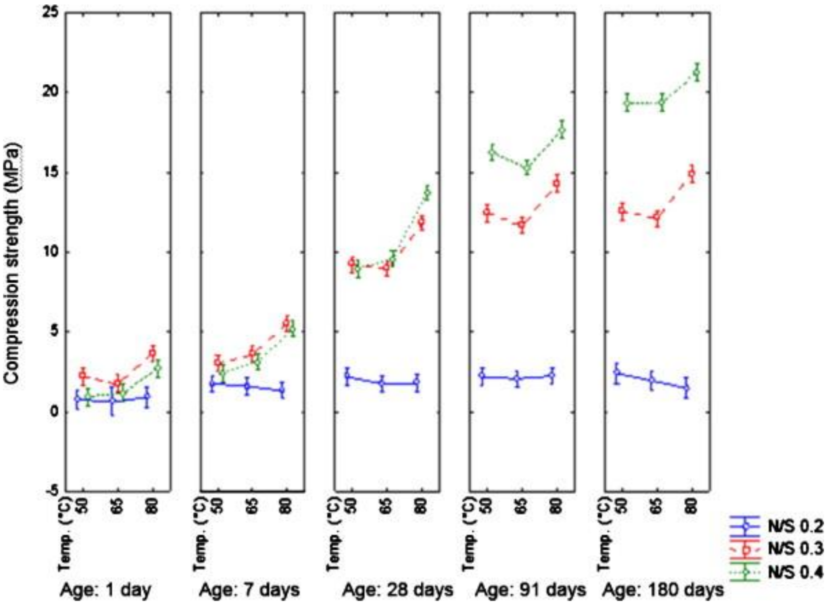


Figure 2.3 Relation between compressive Strength, the temperature for heat curing and duration (Vargas et al., 2011)

Vargas et al. (2011) conducted experiments to find out the influence of the curing temperature and age on the properties of fly ash based geopolymer. In this study, geopolymer paste and mortar samples were kept at the temperature of 50°C, 65°C and 80°C for the first 24 hrs. Geopolymer paste and mortar samples were tested at the age of 1, 7, 28, 91 and 180 days. The ratio of fly ash to sand was kept constant as 1:3 to prepare mortar samples. Compressive strength was relatively low (between 1 MPa and 4 MPa) at the age of 1 day. Mechanical performance of samples produced with the molar ratio (N/S) of 0.3 and 0.4 are different from the samples produced with N/S 0.20. For the sample formed with the ratio of N/S 0.20, mechanical performances changed very little with time. Figure 2.3 shows the effect of the curing temperatures and ages on the compressive strength of geopolymer mortar samples.

Aydin and Baradan (2012) investigated the influence of steam curing at 100°C for 8 hrs and autoclave curingat 210°C under 2.0 MPa pressure for 8 hrs on the mechanical properties of alkali-activated slag mortars. Silicate modulus (M_s) and levels of Na_2O concentration were varied from 0.4 to 1.6 and from 2% to 8% respectively throughout the experiment. In this experiment, the ratio of aggregate to the binder as 2.75 and ratio of water to the binder as 0.44 were kept constant for all mortar mixes. Based on results, autoclaving is observed more effective curing method for low concentrations of Na_2O Solution and low ratio of silicate modulus (M_s). Compressive strength of 70 MPa was obtained for the alkali-activated slag mortars using only 2% Na_2O. Microstructure analysis of the samples indicated the higher ratio of hydrated parts of the slag grains, reduced Ca/Si ratio of C–S–H, reduced pore size distribution of the matrix, and the formation of a stronger aggregate - matrix interface as the main attributes for higher mechanical properties for autoclaving curing compared to the steam curing. They also concluded that drying shrinkage can be reduced significantly with autoclaving curing of the mortar samples.

Li et al. (2013) studied the effects of curing on compressive strength development of calcium-containing geopolymer mortar. The mass ratio of fly ash to sand and the ratio of sodium hydroxide (NaOH) to fly ash were kept as constant as 0.5 and 0.058 respectively. Samples were cured in the following curing regimes: (i) standard curing at 20 ± 3 °C and RH 95%, (ii) steam curing at 60 °C for 24 hrs (iii) steam curing at 60°C for 6 hrs and (iv) oven curing at 60 °C for 24 hrs. After steam curing and oven curing, samples were kept under standard curing until the day of the test. In this work, three types of binder system were used: Class C fly ash, Class F fly ash and Class F fly ash plus Slag. Highest strength at early age was observed for oven curing at 60°C for 24 hrs followed by steam curing at 60°C for 24 hrs. They concluded that the addition of slag to the fly ash showed improvement strength at standard curing. Comparable strength was observed for the samples under standard curing at later ages in their experiment.

Sukmuk et al. (2013) investigated the influence with different temperatures (65°C, 75°C and 85°C) of heat curing for three different durations (24 hrs, 48 hrs, and 72 hrs) on fly ash-clay based geopolymer bricks. From the analysis of results, they proposed a relationship between strength and heat energyfor 7 and 14 days of curing where the heat energy is determined from the product of heat temperature and duration. The proposed relationship, which is shown in Figure 2.4, is given based on sound principle. They also concluded thatvery high temperature and excess duration causes the micro-cracks due to the loss of the pore fluid and as a result, significant reduction in strength was observed.

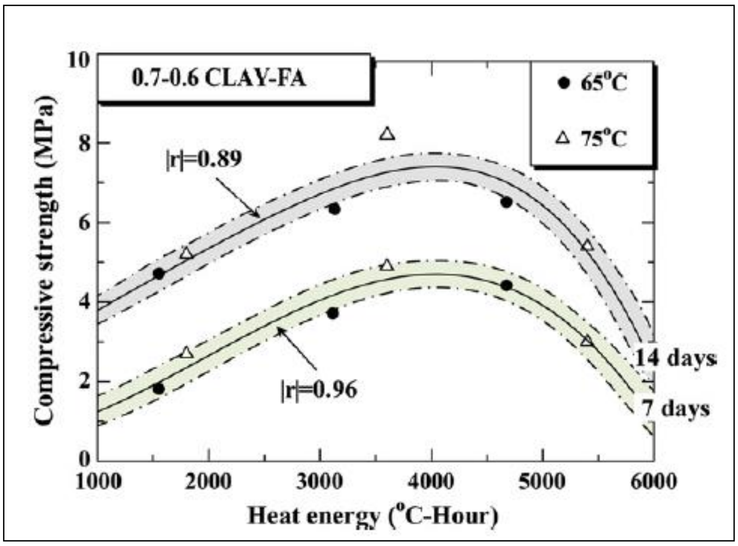


Figure 2.4 Proposed relation between compressive strength and heat energy of geopolymer bricks (Sukmuk et al., 2013)

Gorhan and Kurklu (2014) used two different temperatures (65°C and 85°C) as curing temperature and three different durations (2 hrs, 5 hrs, and 24 hrs) of heat curing of the cast samples in their study to the properties of class-F type geopolymer mortar. This study concluded that curing temperature and curing time had an effect on the physical properties of the geopolymer mortars. The optimal thermal curing temperature was found to be 85°C in this study. They observed that an increase in the curing temperature increased the compressive strength while it did not have a significant effect on the physical properties.

Pangdaeng et al. (2014) studied the influence of different curing conditions on the properties of high calcium fly ash based geopolymer when ordinary Portland cement was used as an additive. 10M sodium hydroxide (NaOH) and sodium silicate solution (15.32% Na_2O, 32.87% SiO_2, and 51.81% H_2O) were used as alkaline solutions throughout the experiment. In this experiment, the ratio of liquid alkaline to binder (L/B) and the ratio of sodium silicate to sodium hydroxide (Na_2SiO_3/NaOH) was maintained constant as 0.40 and 0.67 respectively. 50 mm × 50 mm × 50 mm cube samples fresh geopolymer pastes and mortars were covered with damp cloth and vinyl sheet and kept in 23°C for 24 hrs. After de-molding, samples were kept following different curing conditions: Vapour-proof membrane curing (after de-molding, samples were kept with a vapor-proof membrane wrapping to avoid moisture loss and kept at 23°C), temperature curing (samples cured at 40°C for 24 hours after wrapping with a plastic membrane to avoid moisture loss. After that, the samples were de-molded and kept at 23°C), and wet curing (de-molded samples were kept in water at 23°C until the day of testing). Results indicated high compressive strength at a later age for vapor-proof membrane curing and water curing. High early compressive strength was observed for temperature curing.

Adam and Horianto (2014) conducted experiments on fly ash based geopolymer to find the optimum curing temperature and duration. Mortars sample were cured at different temperature for different duration. 80°C, 100°C and 120°C for 4 hrs, 6 hrs, and 20 hrs were used as curing regimes for the prepared sample in this experiment. In this experiment, the dosage of alkali activator solution was 55% and the ratio of sodium silicate to sodium hydroxide solution was kept constant at1:2. Highest compressive strength was observed for the samples cured at 120°C temperature for 20 hours.

2.3.3 Effect of Different Admixtures in Geopolymer Mortar/Concrete Mixes

Mingyu et al. (2009) conducted experiments on fly ash based geopolymer in which zeolite and bentolite were used as additives. In this experiment, sodium hydroxide (NaOH) solution and present CaO in fly ash were used together as alkali-activator. Results indicated significant role of the concentration of NaOH solution and the CaO content in fly ash on the strength of the geopolymer. Highest strength and best sulfate resistance were observed for the geopolymer materials with zeolite as an additive. Zeolite improves the properties of the fly ash-based geopolymer by involving the geopolymerization process to form a stable zeolitic structure whereas bentonite simply acts as a filler to make up the geopolymer more compacted, but shows no improvement in the compositions and the microstructures of the material.

Khater (2013) investigated the effect of the addition of silica fume on the characterization of geopolymer materials. In this work, geopolymer mix with metakaolin and waste concrete in the ratio of 1:1 (50:50, wt %) was taken as control mix and silica fume was added by 1% to 10% as partial replacement of waste concrete. Results indicated that compressive strength of the geopolymer mixes increased upto 7% addition of silica fume.

Nematollahi and Sanjayan (2014) studied the effects of different superplasticizers on fly ash based geopolymer concrete. In this study, naphthalene (N), Melamine (M) and modified polycarboxylate (PC) based superplasticizers were used at the dosage of 1 % by mass of fly ash. 8M NaOH solution and the ratio of sodium silicate to sodium hydroxide solution 2.5 were used as alkali liquid throughout the study. They concluded that modified polycarboxylate (PC) based superplasticizers was the most effective type resulted in 39% - 45% increase in relative slump whereas N based superplasticizers resulted in 6% - 8% increase in relative slump and M based superplasticizer resulted in 3% decrease in relative slump with reference to the paste without using superplasticizers.

Pangdaeng et al. (2014) used OPC as an additive in their experiment on high calcium fly ash geopolymer mortar. In this experiment, fly ash was replaced by OPC at 0%, 5%, 10%, and 15% by weight of binders. Results show significant reduction of porosities, and water absorption and an increase in the compressive strength. They concluded that formation of C-S-H gel and C-A-S-H gel modified the microstructure of the geopolymer mortar, and generated a dense and strong matrix.

Islam et al. (2014) conducted experiments to find out the possibilities to use of palm oil fuel ash (POFA), ground granulated blast furnace slag (GGBS) and low calcium fly-ash (FA) with manufactured sand (M-sand) to make geopolymer mortar. To investigate the influence of the binders, the content of alkali activators was kept constant and the concentration of sodium hydroxide solution was 12M. The ratio of binder to sand was kept constant as 1:4. The test samples were kept at 65 °C for 24 hrs for heat curing. After the heat curing, specimens were de-molded and left to air-dry condition in the laboratory with the temperature of 27°C and humidity of 70% until the day of the test. This experimental programme shows an increase in compressive strength of geopolymer mortar when the quantity of GGBS in mortar is increased up to 70%. Highest compressive strength was obtained for the geopolymer mortar mix with POFA up to 30% and GGBS.

Ranjbar et al. (2014) investigated the possibility the use of palm oil fuel ash (POFA) in fly ash (FA) based geopolymer mortar. The concentration of the sodium hydroxide (NaOH) solution, the ratio of Na_2SiO_3 solution to NaOH solution and ratio of activator to binder were kept constant as 16M, 2.5 and 0.5 respectively for all the mixes. In this study, fly ash was replaced by POFA ranging from 0% to 100%.To increase the workability of the mortar, additional water was added to the mixes. Casted specimens were covered and kept at a temperature of 65 °C for 24 hours. Using POFA as the replacement of fly ash resulted in a porous geopolymer mortar. Produced geopolymer mortar has shown less density about 6% compared to fly ash based geopolymer mortar. 38% development of the compressive strength between 28 and 112 days for POFA based geopolymer mortar was observed in this study, where the fly ash based mortars gained 97% of their ultimate compressive strength by day 7.

2.3.4 Use of Recycled Aggregates in Geopolymer Mortar/Concrete Mixes

Anuar et al. (2011) have been taken 8M and 14M sodium hydroxide Solutions to finding compressive strength at the age of 3, 7, 14, 21 and 28 days after getting cured in local laboratory ambient condition. The strength of geopolymer concrete based on Waste Paper Sludge Ash (WPSA) incorporated with recycled concrete aggregate (RCA) increased by increasing the molarities of sodium hydroxide (NaOH) solution.

Monish et al. (2012) conducted experiments to determine the performance of concrete mixes in which, demolished waste was used as fine aggregate. The demolished waste collected from the old buildings was considered in this project work. Then the fine aggregates wereextracted and used in concrete making. Concrete specimens with replacements 0%, 10%, 20% and 30% of fine demolition waste were prepared and tested after 7 days and 28 days. From the results, it was shown that replacements for up to 20% of fine demolition waste given almost closer compressive strength to that of conventional concrete. They concluded their research work as recycled aggregate concrete may be an alternative to the conventional concrete. Water required producing the same workability increases with the increase in the percentage of demolished waste and Optimum replacement level of fine aggregate with recycled aggregate is 10%.

Ahamari et al. (2012) studied the feasibility of using ground waste concrete (GWC) powder with class F fly ash to produce geopolymeric binder. In this experimental investigation, 5M and 10M sodium hydroxide solution was used with the ratio of sodium silicate to sodium hydroxide 1 and 2. GWC powder was mixed at levels of 0%, 25%, 50%, 75%, and 100% (by the total mass of GWC powder and FA). Results of this study indicated 50% GWC powder content as the optimum for obtaining the highest compressive strength. SEM/EDX, XRD, and FTIR of produce geopolymeric binders showed the formation of low Calcium semi-crystalline CSH gel which helps to enhance the strength.

Neno et al. (2014) conducted an experimental work to find the performance of mortar mixes, in which the recycled concrete aggregates were used as a replacement to the natural fine aggregate. The particle size distribution was kept constant for both recycled and natural fine aggregate. In this study mortars were cast with 20%, 50% and 100% replacement and the results obtained from these are compared with the reference mortar with no recycled aggregate. Water retentivity, shrinkage, adhesive strength, modulus of elasticity, and water vapor permeability were determined and then, the most satisfactory replacement ratio was determined based on the analysis of the results. Best results in the first stage were observed for 20% and 100% replacement ratios, leading to a careful choice of the 20% ratio for the second stage. Generally, the mortar with 20% replacement ratio performed better than the reference mortar mixes, except for adhesive strength and dimensional stability.

Nuaklong et al. (2015) studied the influences of recycled aggregate on strength and durability properties of fly ash based geopolymer concrete mixes. High calcium based fly ash, sodium silicate solution, sodium hydroxide solution, river sand, recycled concrete aggregate, and crushed limestone coarse aggregate were used to cast the samples. Based on observations, they concluded that recycled concrete aggregate can be used as a coarse aggregate in high calcium fly ash geopolymer concrete mixes. Compressive strength after 7 days was found slightly lower for high calcium fly ash geopolymer concrete mixes than that of the concrete mixes containing crushed limestone. Higher concentration of sodium hydroxide solutions resulted in better performances both in strength and durability properties of geopolymer concrete mixes.

Palankar et al. (2015) conducted a study to examine the performance of GGBS-FA based geopolymer concrete with the usage of weathered steel slag aggregates as the replacement of natural coarse aggregates. The replacement level of natural coarse aggregate by weathered steel slag was adopted as 0%, 25%, 50%, 75% and 100% by volume. The ratio of FA to GGBS was maintained as 0.25:0.75 throughout the study. Na_2O dosage of 5.5% (by weight of binder) with activator modulus of 1.5 and water to binder (w/b)ratio of 0.37 were established by trial mixes to attain concrete of 50 MPa strength and 25-50 mm slump. From this study, they concluded that GGBFS-FA geopolymer mixes with the incorporation of weathered steel slag coarse aggregates were to be observed with slightly lower fresh and mechanical properties but the performance of steel slag as an alternative of coarse aggregates was found to be satisfactory for the structural and pavement application.

Mithun et al. (2015) attempted to investigate the fatigue characteristics of alkali-activated slag concrete mixes incorporating copper slag as the alternative of natural fine aggregates (river sand). To prepare the concrete mixes, activator modulus, water to binder ratio and Na_2O dosage were considered as 1.25, 0.4 and 4% respectively. Copper slag in mixes was varied by 0%, 50%, and 100% by volume of river sand. In this study, slightly better fatigueperformance was observed for the alkali-activated slag concrete having copper slag as fine aggregates compared to the alkali-activated concrete mixes with river sand due to the denseanduniform interfacial transition zone.

Palankar et al. (2016) also conducted durability performance of alkali-activatedslag concrete (AASC) and alkali activated slag fly ash concrete (AASFC) incorporating steel slag as the replacement of natural coarse aggregate. In this study, the replacement levels were chosen as 0%, 50%, and 100%. Compressive strength in the range 55 ± 5 MPa after 28days of curing and with a workability of 25-50 mm were targeted to obtain and a binder content of 425 kg/m^3 and water/binder ratio of 0.40 were selected. Na_2O dosages were varied also from 4% to 5.5% (by total binder) with different ratio GGBS to fly ash. They concluded that the inclusion of steelslag aggregates slightly reduced the durability performance of AASC and AASFC mixes compared to ordinary Portland cement concrete (OPCC). They also found out AASC and AASFC with steel slag aggregates as eco-friendly having lower energy requirement and lower production cost ascompared to OPCC.

Mithun and Narasimhan (2016) studied the properties of alkali-activated slag concrete using copper slag as the replacement of natural fine aggregates. Copper slag in mixes was varied by 25%, 50%, 75% and 100% by volume of river sand. To prepare the concrete mixes, activator modulus and Na_2O dosage were considered as 1.25 and 4% respectively. Similar modulus of elasticity, lower totalporosity, lesser water absorption, and reduced chloride ion penetration were observed in this study for alkali-activated slag concrete with sand or copper slag as compared to ordinary Portland cement concrete.

Vásquez et al. (2016) investigated the synthesis of geopolymers based on alkaline activation of concrete demolition waste (CDW) using sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) as alkaline activators. In their study, Portland cement and metakaolin were used upto 30% by weight to CDW to produce binary and hybrid geopolymer paste. CDW powder was generated from the demolition waste passing through the hammer mill first and then through a ball mill. Geopolymers activated with sodium silicate solution showed the highest compressive strength at room temperature. Maximum compressive strength was obtained 25 MPa for geopolymers consists of 100% CDW whereas geopolymer consist of CDW + 30% OPC gained 33 MPa at 28 days at room temperature curing and geopolymers with CDW + 10% MK, reached 46.4 MPa.

Whiting et al. (2012) found the effect 100% recycled concrete aggregates on the drying shrinkage properties of the concrete. Prepared samples were tested at the age of 112 days to determine drying shrinkage. In this study, fly ash also was incorporated. Recycled aggregate concrete mix without fly ash was observed to have 25% average higher drying shrinkagethan the control mix whereas mix with the fly ash had only 7%increase.

Eguchi et al. (2003) discussed the mechanism of drying shrinkage and water loss of concrete having recycled coarse aggregates. From their experimentalwork, higher drying shrinkage of concrete mixes having recycled coarse aggregates was observed than that of natural aggregate concrete.

Domingo-Cabo et al. (2009) conducted experimental research to determine the creep and shrinkage properties of recycled aggregate concrete. Recycled aggregates generated from construction waste and demolitions of concrete work were incorporated at 20%, 50%, and 100%. Similar variation in shrinkage and creep properties was observed for both recycled aggregate concrete and conventional concrete. After a period of 180 days, an increase in the deformations by creep of 51% and by shrinkage of 70% was observed for the concrete mixes having 100% recycled aggregates as compared to the control mix.

2.4 SUMMARY

Numerous research studies on fly ash based and slag based geopolymer mortar have been carried out in recent years. Most of the researchers have focused on the physical, mechanical and microstructural properties of the geopolymer mortar varying the following parameters: concentration of sodium hydroxide (SH) solution, ratio of sodium silicate (SS) solution to SH solution, temperature for heat curing, duration of heat curing, ratio to the alkaline liquid to fly ash/slag etc. Based on the literature, alkaline liquid consisting of SS and SH solution is found to be the most effective to produce geopolymer matrix with proper mechanical properties. Literature indicates an increase in compressive strength of geopolymer with increments of theconcentration of SH solution. Concentration of SH solution varying from 6M to 14M is observed to be the best range to get higher compressive strengths. Range of 75°C - 90°C has been found to be beneficial for heat curing of geopolymer samples. It has been seen that recycled fine aggregate can be used in the replacement of natural fine aggregates for getting desired properties of concrete.

Most of the researchers have conducted their experiments on geopolymer mortar/concrete usingmixes of natural sand and natural coarse aggregate. Continuous research efforts are being made to find alternative materials for the replacement of natural aggregates. Few researchers investigated the properties of geopolymer concrete incorporating recycled coarse aggregate as a partial/full replacement of natural coarse aggregate. Experiments are conductedto find the properties of OPC mortar mixes produced with recycled fine aggregate by many researchers. But there is no significant research on the use of recycled fine aggregate generated from C&DW to produce geopolymer mortar mixes. Incorporation of recycled fine aggregate in geopolymer mortar mixes will be advantageous to promote sustainability in the construction industry as two industrial by-product materials fly ash (from the thermal power plant) and recycled fine aggregate from C&DW are going to be used. Such use of recycled aggregate and fly ash also helps to manage effectively, the land depletion problems arising out dumping those waste materials, which helps to reduce environmental and ecological problems. Considering these problems and solutions both, the aim and objectives for this research work are framed and stated in the following.

2.5 AIM AND OBJECTIVES

This study aims to use alternative materials, geopolymer as a binder and recycled aggregate effectively to minimize the environmental problems generated from the dumping of the fly ash and C&DW, especially concrete wastes, and to reduce the use of the virgin natural resource. This can be achieved by recycling these by-product/waste materials as a partial/full replacement to cement and river sand to produce geopolymer mortar mixes, which can be used for various purposes and promoting sustainability in the construction industry. To achieve the aim of our research, the objectives for the present work are set up as followings.

2.5.1 Objectives

- To produce fly ash based geopolymer mortar mixes using recycled fine aggregate as partial replacement of natural river sand and to study the properties (workability, compressive strength, and drying shrinkage) of the geopolymer mortar mixes made of recycled fine aggregate.

- To investigate the effects of the following parameters on the properties of the geopolymer mortar mixes.
    - Concentration of sodium hydroxide (SH) solution.
    - Ratio of sodium silicate (SS) solution to sodium hydroxide (SH) solution in the alkaline liquid (AL).
    - Curing regime
    - Alkaline liquid to fly ash ratio.
- To compare the properties of geopolymer mortar mixes produced with recycled fine aggregate with the properties of mixes produced with locally available sand.
- Improvement of the properties of fly ash based geopolymer binder with the incorporation of ground granulated blast furnace slag (GGBFS).
- To develop a model to predict the properties of geopolymer mortar mixes using an artificial neural network.

2.6 SCOPE OF WORK

In order to achieve the objectives of the study, the experimental study was conducted in the following phases:

- Generation of recycled fine aggregate from the available tested concrete specimens i.e. cubes, cylinders, and beams without reinforcement in the laboratory.
- Determination of the physical properties of recycled fine aggregate and compare with the guidelines given by Indian standards.
- Study the properties of fly ash based geopolymer mortar mixes using recycled fine aggregate varying above mentioned parameters.
- Improvement of the properties of fly ash based geopolymer binder.
- Using the experimental data, creation of a model to predict the properties of geopolymer mortar mixes using neural network.

To find out the effect of different parameters on the properties of geopolymer mortar mixes having RFA partially, concentration of SH solution was varied from 6M to 16M, ratio of SS solution to SH solution was varied from 1.0 to 2.0 and alkaline liquid to fly ash ratio was varied from 0.4 to 0.8. Cast samples werekept under three curing regimes namely ambient temperature, water and heat curing at 80°C for 24 hoursin this experimental programme. After the heat curing, samples were kept into the ambient air condition until the test.

Chapter 3

MATERIALS AND METHODOLOGY

3.1 INTRODUCTION

The details of the experimental programme in terms of materials, the experimental setup for determining the effect of different parameters and the testing procedures have been discussed in this chapter in order to achieve the objectives of this research work.

3.2 MATERIALS

Class F fly ash, locally available sand, recycled fine aggregate (RFA) from concrete waste, alkaline liquid (combination of sodium hydroxide solution and sodium silicate solution), and water were used as materials for the preparation of geopolymer mortarsamples.

3.2.1 Fly Ash (FA)

Fly ash (FA) is a by-product material from thermal power plants resulting from the combustion of pulverized coal in the coal-fired furnaces. Two types of fly ash are available namely, Class C Fly Ash and Class F Fly ash.

Class C Fly ash: This type of fly ash is normally produced by burning lignite or sub-bituminous coal. This type of fly ash may have CaO content more than 10%. In addition to pozzolanic properties, it also possesses cementitious properties.

Table 3.1 Properties of fly ash and GGBFS

Properties	**Obtained Value**	
Materials	**Fly ash**	**GGBFS**
Specific Gravity	2.2	2.9
Blaine's Fineness (cm^2/gm)	2161.86	3570

Class F Fly ash: This type of fly ash is normally produced by burning anthracite or bituminous coal. This type of fly ash usually has CaO content of less than 5%. It has pozzolanic properties only. For the present work, class F type of fly ash was used.

Properties of fly ash used in this work are tabulated in Table 3.1 and Table 3.2 represents the details of chemical composition.

Table 3.2 Chemical Composition of Fly ash and GGBFS (Percentage by weight)

Constituents	Fly Ash	GGBFS
CaO	0.73	34.07
Al_2O_3	32.24	16.98
Fe_2O_3	2.84	1.26
SiO_2	58.90	32.57
MgO	0.89	9.69
Na_2O	0.35	0.20
K_2O	1.12	0.08
SO_3	0.50	0.84
Insoluble Residue	2.28	4.01
Loss of Ignition	0.03	0.02

3.2.2 Ground Granulated Blast Furnace Slag (GGBFS)

Ground-granulated blast furnace slag (GGBFS) is also a by-product material fromiron and steel production, which is obtained by quenching molten iron slag from a blast furnace in water or steam. After quenching, granular product used to be dried and ground into a fine powder and the process produce a glassy type of material. The chemical composition of GGBFS used in this work is shown in Table 3.2. GGBFS was incorporated at 10%, 20%, 30%, 40%, and 50% level to the total binders to improve the properties of geopolymer binder.

3.2.3 Aggregates

Quality of aggregates plays an important role to attain proper strength and durability of mortar mixes. Aggregates normally are taken as inert material, but aggregates are not truly inert and its physical, thermal, and sometimes also chemical properties influence the performance of concrete. Aggregates are normally divided into two types - fine aggregate and coarse aggregate. In the present study, natural fine aggregate (locally procured river sand), and recycled fine aggregate (generated from old tested specimens) were used to prepare the mortar samples.

3.2.3.1 Natural Fine Aggregate (NFA)

The aggregate material, which passes through 4.75 mm IS sieve and retained on 150 microns IS sieve is termed as fine aggregate. Usually, locally procured river sand is used as natural fine aggregate. The NFA used for the present experimental works was locally procured river sand and it conformed to grading zone II (IS: 383 - 2016). The physical properties are provided in Table 3.3. The particle size distribution of fine aggregate is shown in Figure 3.2.

3.2.3.2 Recycled Fine Aggregate (RFA)

Tested concrete specimens e.g. cubes, cylinders, beams etc. without reinforcements were used as the source of recycled fine aggregate (RFA). To make RFA, the specimens without reinforcement were manually broken down into small pieces to the size of 40 mm to 20 mm. Then these fractions from 40 mm to 20 mm of particles were placed in jaw crushers to make it finer. The crushed pieces of concrete were then separated into different fractions depending on their size. The fraction passing through 4.75 mm sieve and retained on 150 microns was taken as RFA for the preparation of samples. Figure 3.1 shows the final product RFA from tested concrete specimens. In the present study, natural fine aggregate was replaced by RFA at the rate of 10%, 20%, 30%, 40%, and 50%.

Figure 3.1 Formation of RFA from tested concrete Specimens

The properties of NFA and RFA is shown in Table 3.3 and the particle size distribution of natural fine aggregate (river sand), recycled fine aggregate and different combination of RFA and sand are shown in Figure 3.2.From this particle distribution curve, it has been observed that all the combination of RFA and sand, which were used to produce geopolymer mortar mixes, were conforming to zone II as per the guidelines given by IS: 383 - 2016, whereas locally available sand and RFA conformed individually to zone II and zone I respectively.

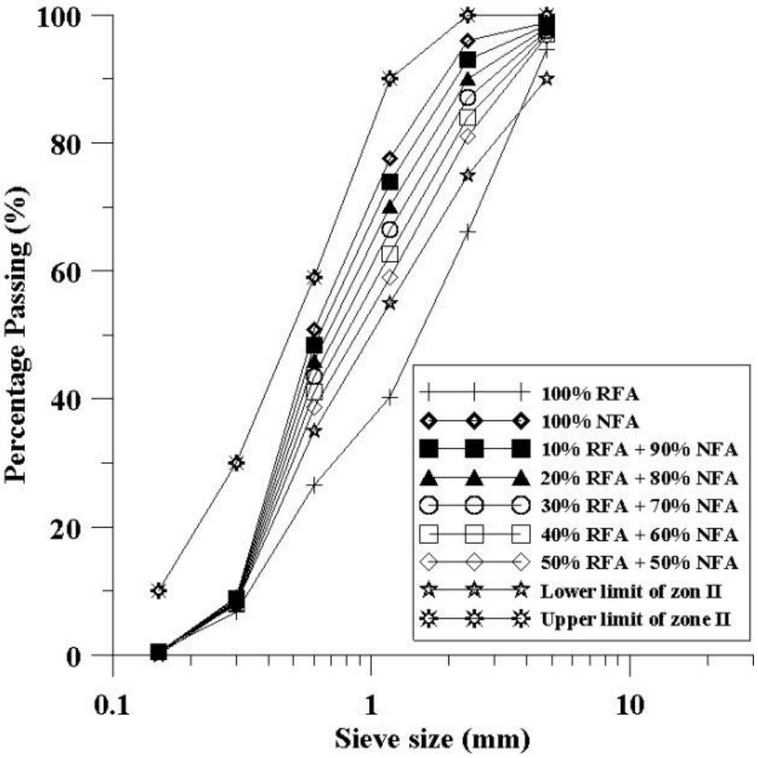


Figure 3.2 Grain size distribution curve

Table 3.3 Properties of NFA and RFA

Characteristics		**Obtained Values**	
Type		Sand	RFA
Specific Gravity		2.66	2.32
Bulk Density (gm/cc)	Loose	1.417	1.287
	Compacted	1.683	1.544
Grading Zone		II	I
Water Absorption		1 %	9.2%

3.2.4 Alkaline Liquid (AL)

A combination of sodium silicate (SS) solution and sodium hydroxide (SH) solution was chosen as the alkaline liquid (AL). The SS is available commercially in solution form. The chemical composition of SS solution was Na_2O - 8.5%, SiO_2 - 28.0% and water - 63.5% by mass. The SH is available commercially in flakes or pellets form. For the present study, SH flakes with 98% purity were used for the preparation of SH solution. After the preparation of SH Solution, heat was liberated and it was allowed to cool down before mixing with SS solution in order to prepare AL. The chemicals required for the present study was procured from local dealer/supplier. The AL used to be prepared before 24 hours prior to use in the mixes.

3.2.4.1 Preparation of alkaline liquid

AL, a mixture of SH solution and SS solution can be prepared by different approaches i.e. based on the concentration of SH solution, based on Na_2O content, based on activator modulus etc. In the present study, AL was prepared based on the concentration of SH solution and the ratio of SS solution to SH solution. Concentration of SH solution was varied from 6M to 16M and the ratio of SS solution to SH solution was varied from 1.0 to 2.5. For the preparation of AL, solution of SH was prepared first based on the required concentration and then the liquid SS solution was mixed with the prepared SH solution according to the desired ratio by mass only.

Molarity is a unit of concentration measuring the number of moles of a solute per litre of solution. In order to prepare one litre of 1M solution, one mole of solute is required to be dissolved into water. Therefore, to prepare 1M SH solution, 40g SH pellets/flakes is required to dissolve into the water for onelitre SH solution. As SH solution was prepared and taken by mass, the mass of SH pellets/flakes required for one kg of the SH solution with the desired concentration needs to be determined. The following procedures were followed to determine the mass of SH pellets/flakes for the preparation of one kg solution with the desired concentration.

- The required quantity of SH pellets was weighed and taken into glass flask having the mark of one litre. For examples, if we need to prepare 8M SH solution, we need to take 320g (8 × 40g) of SH pellets.
- Then water is slowly poured into the flask upto the mark of one litre and stirred properly. As the reaction between sodium hydroxide and water is exothermic, heat will be generated. As a result, little quantity of water used to be evaporated. Evaporated quantity of water was added after the solution got cooled down. Then, the weight of one-litre solution was noted down. Based on this weight of the SH solution, quantity of SH pellets are calculated to produce one kg of SH solution for different concentration of the solution. Calculated quantities of SH pellets required to produce one kg solution for different concentration solution is given in Table 3.4. Similar procedure was adopted by Hardijto and Rangan (2005) in their study.
- SS solution is available in liquid form commercially. Based on the ratio of SS solution to SH solution, the required quantity of SS solution was weighed and taken to mix with the prepared SH solution.

Table 3.4 Required sodium hydroxide pellets/flakes per kg solution

Concentration of SH (M)	**SH pellets required for one kg solution (g)**
6	206
8	262
10	314
12	362
14	404
16	444

3.2.5 Water

Water is an important ingredient of concrete as it actively participates in the chemical reaction with binder. Since it helps to form the strength attributing products, the quantity, and quality of water is required to be looked into very carefully. In the present investigation, distilled water was used for the preparation of SH solution and tap water was used for the curing purposes.

3.3 METHODOLOGY

3.3.1 Casting of Specimens

In batching, first weighing of all the materials was done. Then, first NFA/RFA and fly ash were added and were dry mixed for 2 - 3 minutes. Then, the AL was added and again mixed for 2 - 5 minutes. The mixture was now ready for pouring in the molds. Cubes having dimension 70.7 mm × 70.7 mm × 70.7 mm were cast to study the water absorption capacity, compressive strength and prism specimens having dimension 25 mm × 25 mm × 285 mm were cast to determine drying shrinkage of various geopolymer mortar mixes. The cubes and prism molds were filled with fresh mortar and compaction was done by hand operations and then using a vibrating table. Figure 3.3 (a - d) represents the cast samples and de-molded samples prepared from geopolymer mortar mixes.

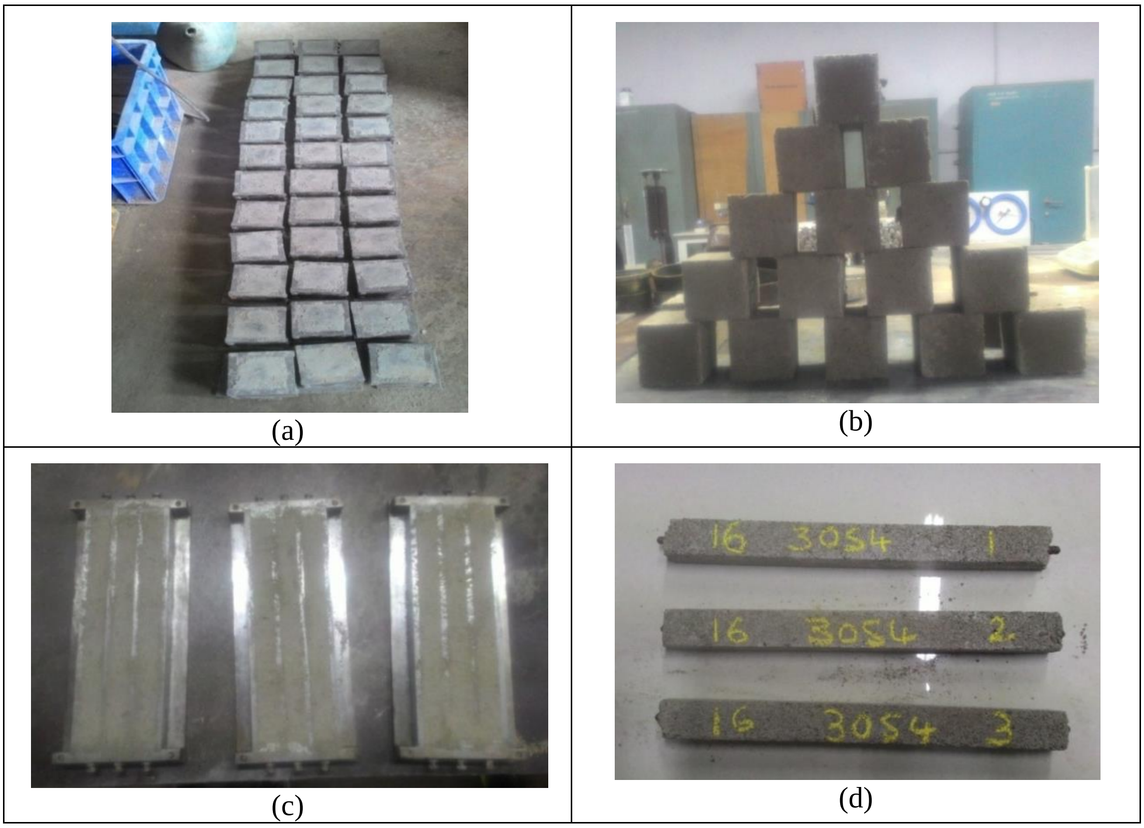

(a) (b) (c) (d)

Figure 3.3 (a) Cast cube samples; (b)De-molded cube samples; (c) Cast prism specimens; (d) Prism samples after de-molding

3.3.2 De-Molding of Test Specimens

The test specimens were removed from the molds after 24 hours (after attaining the initial strength), while de-molding the specimens, care was taken such that there would not be any damage to the specimens. Especially, prism specimens used to get broken into pieces if de-molding were done carefully.

3.3.3 Curing of Test Specimens

After careful de-molding, the cubes were kept under different curing regimes. One set of the samples was kept in the water chamber, which is filled with normal tap water at the room temperature. One set of the samples was kept for ambient air curing. Another set of samples was kept in an oven at 80°C temperature for heat curing for 24 hours. After heat curing, samples were kept at room temperature until the day of testing. All cured cube samples were tested at the age of 3 days, 7 days, 28 days, and 56 days to determine compressive strength of different fly ash geopolymer mortar mixes. change of length in longer dimension of cast prism specimens was noted down at the age of 3 days, 7 days, 28 days, 56 days, 90 days, 120 days, 150 days, and 180 days.

3.4 TESTS ON SPECIMENS

3.4.1 Flow Test

Workability of mortar mixes is its ease of use measured by the flow of the mortar mixes. The standard flow tests use a standard conical frustum shape with a base diameter of 100 mm. This mortar sample is placed on a flow table and dropped 25 times within 15 seconds. As the mortar is dropped, it spreads out on the flow table. The initial and final diameters of the mortar sample are used to note down for the calculation of observed flow. Flow is defined as the increase in diameter of the mortar mixes divided by the original diameter of the mortar mixes multiplied by 100. Flow table tests, to determine the workability of geopolymer mortar mixes, were done according to the guidelines given by IS: 5512 - 1983.

3.4.2 Water Absorption Test

Water absorption test was carried out to determine the absorption of water by the geopolymer mortar mixes produced with RFA at a different percentage level. After de-molding, the cubes having 50 cm^2 surface area were used to determine the water absorption capacity according to the guidelines given by ASTM standard C642 - 13.

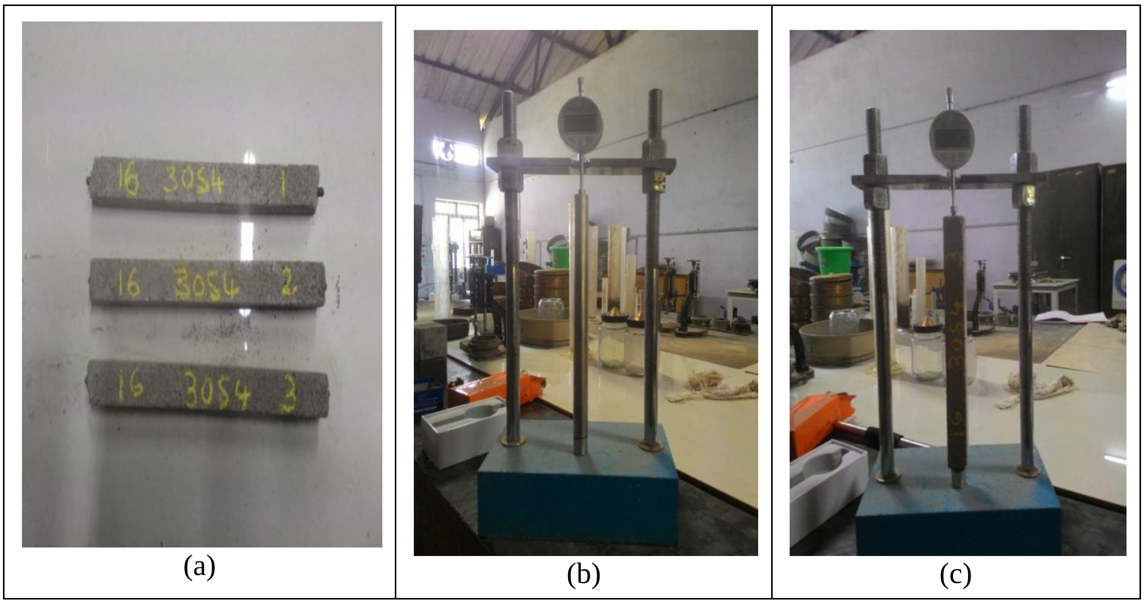
3.4.3 Compressive Strength Test

The compressive strength of concrete is given in terms of the characteristic compressive strength for the cubes tested at the different age of curing. The compressive strength test was conducted on cast cubes at 3 days, 7 days, 28 days, and 56 days of curing under different regimes. At a particular duration, the average strength of three test cube samples cast with different of geopolymer mortar mixes was taken as the compressive strength of the respective fly ash based geopolymer mortar mixes having RFA partially. Figure 3.5 shows the failure pattern of the cube samples cast with fly ash based geopolymer mortar mix having RFA partially.

Figure 3.4 Failure pattern of cube samples after compression test

3.4.4 Drying Shrinkage Test

Drying shrinkage of the cast samples was measured in terms of length change in longer direction due to the loss of moisture from the samples. Length of each prism specimen was measured using a length comparator immediately after de-molding. After that, all samples were kept under desired curing regimes. Then, the length of specimens was measured for getting change in length at different ages of 3 days, 7 days, 28 days, 56 days, 90 days, 120 days, 150 days and 180 days. In this study, the observed value of drying shrinkage of the various mortar mixes has been expressed as micro-strain. Figure 3.3 (a-c) shows the cast samples, calibration of length comparator using a standard bar and measurement of the change of length of the prism samples.



(a) (b) (c)

Figure 3.5 (a) Cast Prism Samples (b) Calibration of length comparator using standard bar (c) Measurement of change of length of cast samples

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter describes the results of the test program designed to study the properties of the various fly ash based geopolymer mortar mixes using RFA partially as described in the previous chapter. Effect of different parameters on workability, water absorption capacity, compressive strength, and drying shrinkage of different geopolymer mortar mixes are discussed in the following sections.

4.2 WORKABILITY

Flow table test, to determine the workability of geopolymer mortar mixes, was conducted according to the guidelines given by IS: 5512 - 1983. Usually, FA based geopolymer mortar mixes were sticky type in nature. From the experimental results, it has been observed that geopolymer mortar mixes produced with AL/FA=0.4 were most harsh mixes and mixes produced with AL/FA = 0.8 were more workable mortar. Figure 4.1 - 4.3 represent the pictorial view of flow observed in the laboratory for different AL/FA. From the figures, it can be easily identified that collapses occurred for the mixes produced with AL/FA = 0.4 and highly flowable mortar mixes with AL/FA=0.8. But mortar mixes produced with AL/FA = 0.8 had problems on setting time. These mixes required more time to set and also during de-molding of cube/prism samples, it was very difficult to get the samples in proper shape and dimensions. Therefore, it is better to avoid using the AL/FA = 0.8 to produce FA based geopolymer mortar and AL/FA = 0.8 ratio has been discarded for the further study.

Mortar mixes were observed to be stiffer in condition when the replacement of NFA by RFA was increased to produce the mortar mixes. Mortar mixes with 50% RFA were found to bestiffer in condition. It may be due to the high water absorption capacity of RFA. RFA may absorb the water molecules from the AL due to its porous nature. As a result, the mortar mixes became stiffer.

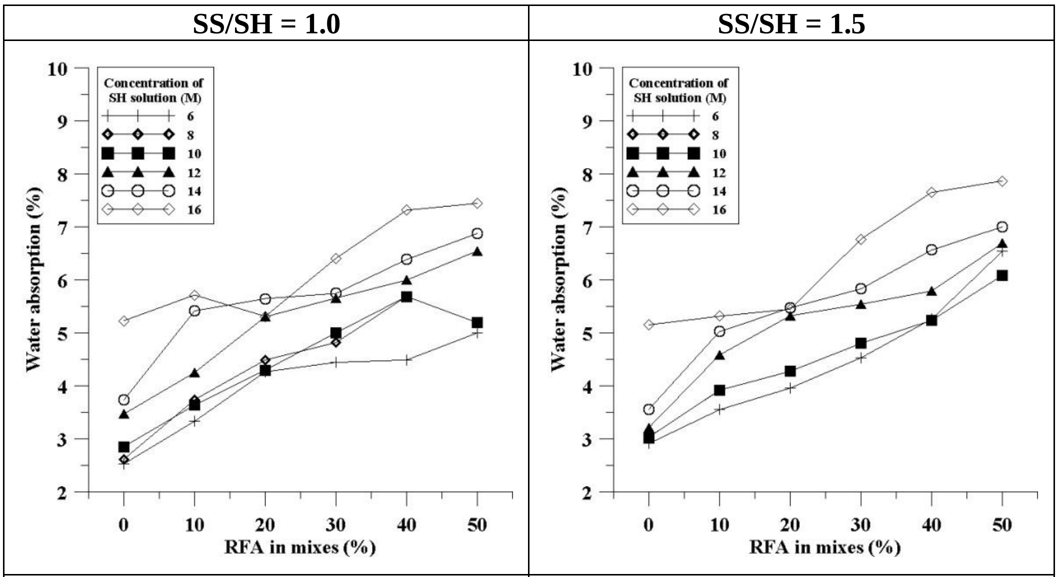
Figure 4.1 Flow for the mixes produced with AL/FA = 0.4

Figure 4.2 Flow for the mixes produced with AL/FA = 0.6

Figure 4.3 Flow for the mixes produced with AL/FA = 0.8

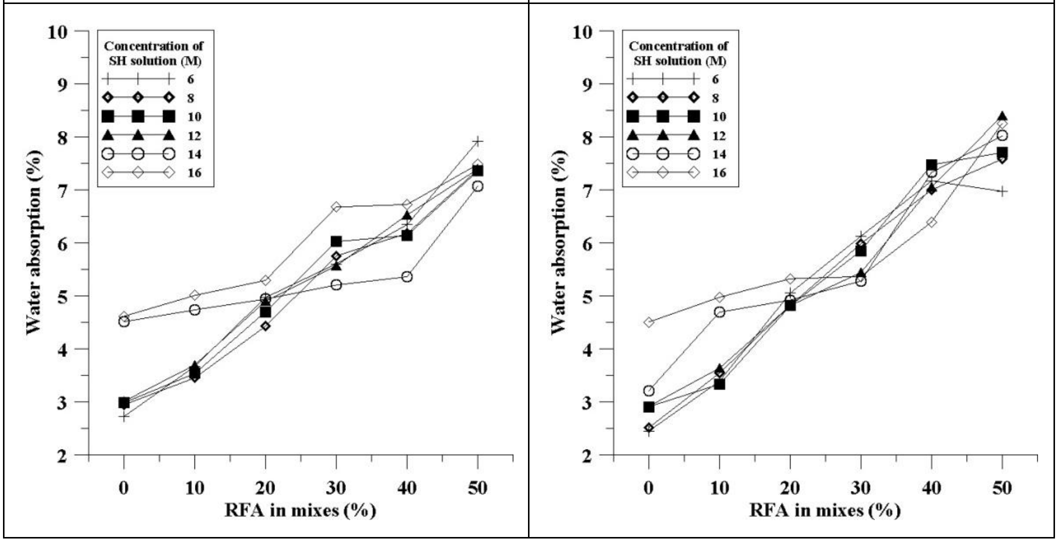
4.3 WATER ABSORPTION CAPACITY

To determine the water absorption capacity of geopolymer mortar mixes of each batch, the guidelines given by the ASTM standard C 642 - 13 was followed and the water absorption capacity of different geopolymer mortar mixes was calculated. In the following, effects of alkaline liquid and RFA content in the mixes on water absorption capacity of the produced fly ash based geopolymer mortar mixes have been discussed. Highest water absorption capacity was observed for the mortar mixes produced with the consideration of 16M SH solution, AL/FA = 0.4 and SS/SH = 2.5.Figure 4.4 and Figure 4.5 shows the variation of water absorption capacity of geopolymer mortar mixes produced with AL/FA=0.4 and AL/FA = 0.6 respectively.



SS/SH = 1.0

SS/SH = 1.5



SS/SH = 2.0

SS/SH = 2.5

Figure 4.4 Water absorption capacity of geopolymer mortar produced with AL/FA=0.4

4.3.1 Effect of SH solution

Higher water absorption capacity of FAbased geopolymer mortar mixes was observed for the mortar mixes produced with the AL having higher concentration of SH solution. To prepare higher concentration of SH solution, more quantity of SH pellets is required and the quantity of water requiredis less. Therefore, total water quantity in AL is less when the higher concentration of SH solution was adopted to prepare AL. Even though higher concentration of SH solution in AL helps to form silico-aluminate structures more than that of less concentration of SH solution in AL, due to the porous nature of RFA, geopolymer mortar mixes with RFA may show higher water absorption capacity when higher concentration of SH solution was considered to prepare AL.

4.3.2 Effect of AL/FA ratio

From the Figure 4.4 and Figure 4.5, it can be concluded that water absorption capacity of FA based geopolymer mortar mixes produced with AL/FA = 0.6 is significantly lower than that of mortar mixes produced with AL/FA = 0.4. When higher AL/FA ratio was adopted to produce geopolymer mortar mixes, then sufficient amount of AL was available to react with FA to form dense geopolymeric matrix. Therefore, mortar mixes produced with higher AL/FA ratio showed lower water absorption capacity.

4.3.3 Effect of SS/SH ratio

Most of the geopolymer mortar mixes exhibited higher water absorption capacity when higher SS/SH ratio was adopted to prepare the AL and used to produce mortar mixes. But for all concentration of SH solution, the similar trend of the variation of water absorption capacity of mortar mixes was observed.

4.3.4 Effectof RFA content

As the percentage level of NFA by RFA is higher, water absorption capacity of geopolymer mortar mixes was observed to be higher. Higher water absorption capacity of RFA itself may be the reason for the higher water absorption of the geopolymer mortar mixes having higher quantity of RFA.

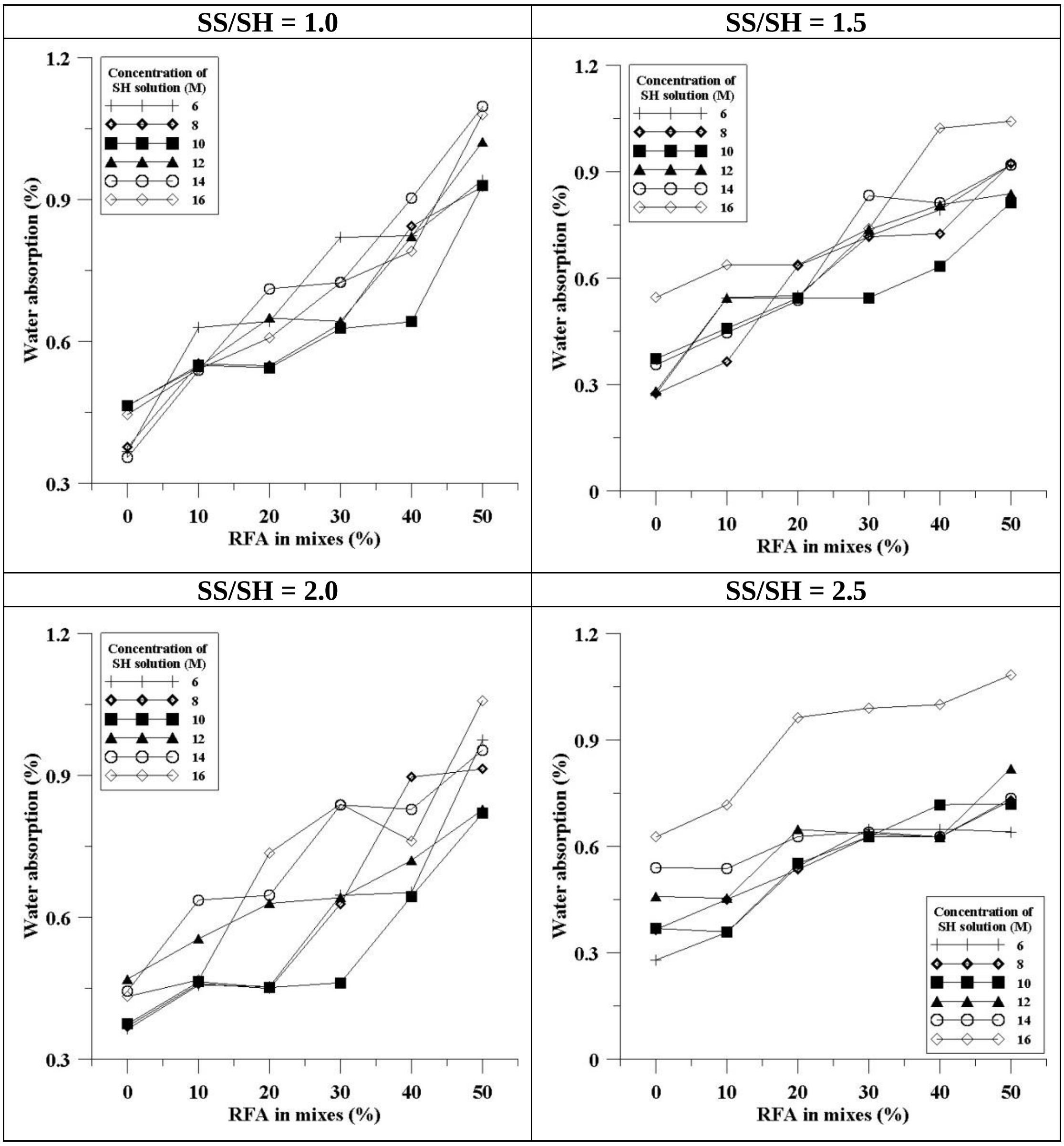


Figure 4.5 Water Absorption capacity of geopolymer mortar produced with AL/FA=0.6

4.4 COMPRESSIVE STRENGTH

Compressive strength of cast cube samples having surface area of 50 cm^2 was determined at the age of 3 days, 7 days, 28 days and 56 days of different curing regimes. Cast cube samples after de-molding were kept for curing in following conditions.

- Ambient temperature curing

- Water curing
- Heat curing at 80°C for 24 hours and then ambient temperature curing.

In the following sections, compressive strength at 3 days, 7 days, 28 days and 56 days for different curing regime are presented and discussed the effects of different parameters on compressive strength of mortar mixes.

4.4.1 Ambient Temperature Curing

With the ambient temperature curing for 3 days, 7 days, 28 days and 56 days, the compressive strengthof the mortar mixes produced with the consideration of AL/FA = 0.4 and 0.6 are shown in Figure 4.6 - 4.9 and Figure 4.10 - 4.13 respectively. According to ASTM C270, mortar mixes have been categorized into different categories based on compressive strength and mentioned suitable activities to use the different types of mortar effectively. Table 4.1 and Table 4.2 represent the different categories and their possible usage given by ASTM C270.

Table 4.1 Categorization of mortar mixes

Type	**Minimum average compressive strength at 28 days (MPa)**
M	17.2
N	12.4
S	5.2
O	2.4

As per obtained result for ambient temperature cured samples, most of the mortar mixes produced with AL/FA ratio 0.4 can be categorized as "S" type of mortar mixes. But few mixes are there of "O" type of mortar mixes. Most of the mortar mixes produced with AL/FA ratio 0.6 can be categorized as either "M" or "N" type of mortar mixes. But very few mixes are there of "S" type of mortar mixes.

Table 4.2 Selection of mortar type

Building Segment	**Type of Mortar**
Exterior, above grade:	
Load-bearing	**N or S or M**
Non load-bearing	**N**
Parapet wall	**N or S**
Exterior, at or below grade	**S or M**
Interior:	
Load-bearing	**N or S**
Non load-bearing	**N**

According to IS: 2250-1981, grade of masonry mortars has been given based on the 28 days compressive strength in Table 4.3. Based on compressive strength at 28 days of mortars, the different grades are shown in table 4.3.From the obtained results in this study, it has been observed that mortar cube samples cured at ambient temperature are falling into the category of MM3 to MM7.5 for the mixes with both AL/FA ratios. IS: 2250-1981(sec 7.1.1 to sec 7.1.6) also gives the guidelines to select the appropriate grade of mortars for different type of works.

Table 4.3 Grade of Masonry Mortar as per IS: 2250-1981

Compressive Strength at 28 days (MPa)	**Grade of Mortars**
0.5 to 0.7	MM0.5
0.7 to1.5	MM0.7
1.5 to 2	MM1.5
2 to 3	MM2
3 to 5	MM3
5 to 7.5	MM5
7.5 and above	MM7.5

4.4.1.1 Effect of the concentration of SH solution

For AL/FA = 0.4, compressive strength of geopolymer mortar cubes cured atambient temperature was being noticed to be increased with the increase of the concentration of SH solution in AL. Geopolymer mortar mixes produced with 14M SH solution after 3 days curing showed the higher compressive strength. But after 7 days, 28 days and 56 days of ambient temperature curing, mixes produced with 16M SH solution exhibited higher compressive strength.

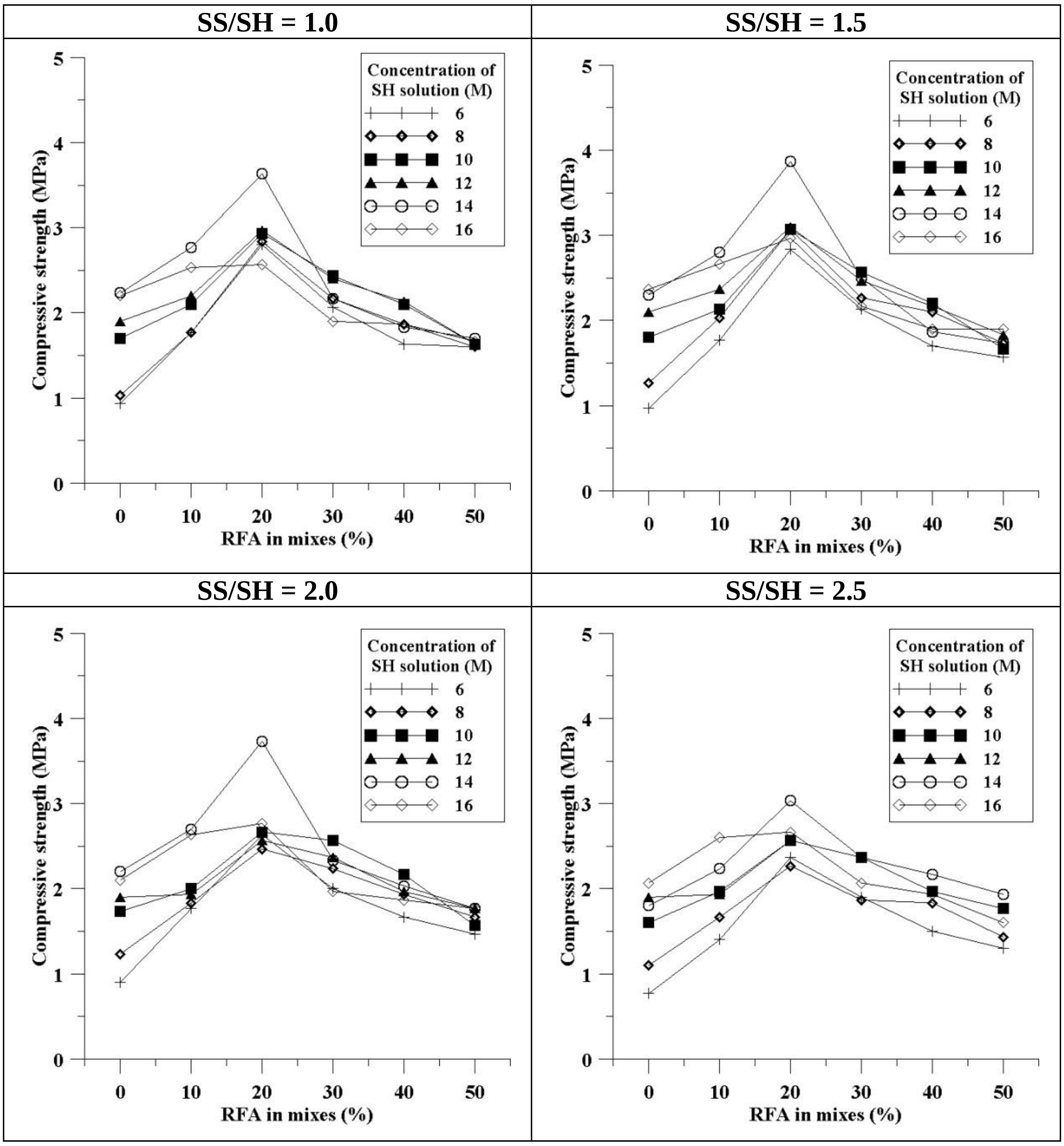


Figure 4.6 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 3 days air curing

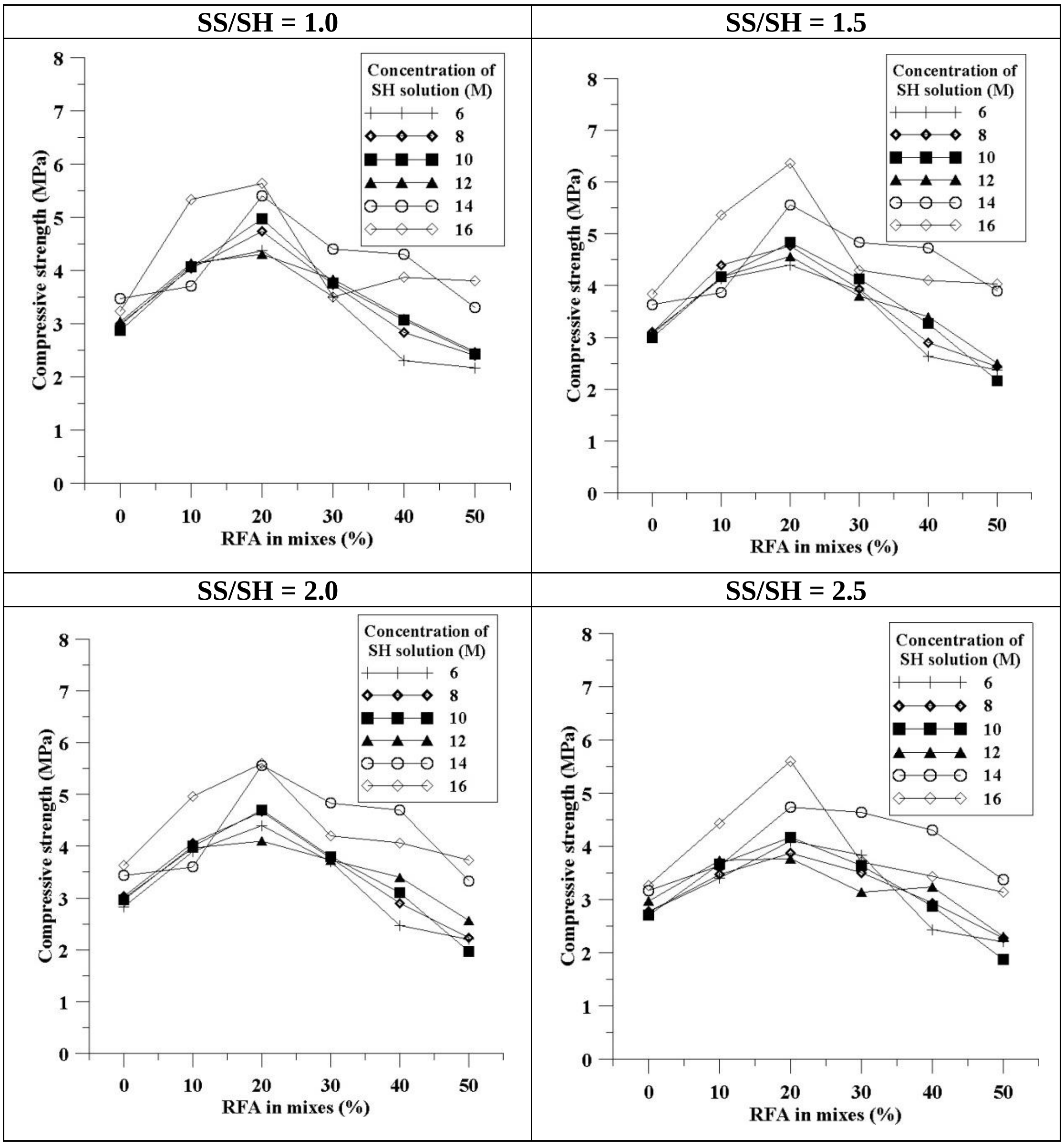


Figure 4.7 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 7 days air curing

For AL/FA = 0.6, mortar mixes produced with the AL having higher concentration of SH solution showed better strength. Mortar mixes with 16M SH solution exhibited higher strength after 3 days, 7 days and 28 days of ambient curing. But after 56 days, mixes with 14M showed the highest strength. Higher concentration of SH solution in AL helps the reaction to occur between FA and AL for the formation of geopolymeric structures to achieve higher compressive strength. Studies by Ridtirud et al. (2011), Somna et al. (2011), Budh and Warhade (2014), Gorhan and Kurklu (2014) also indicated that higher compressive strength was achieved with higher concentration of SH solution.

4.4.1.2 Effect of SS/SH ratio

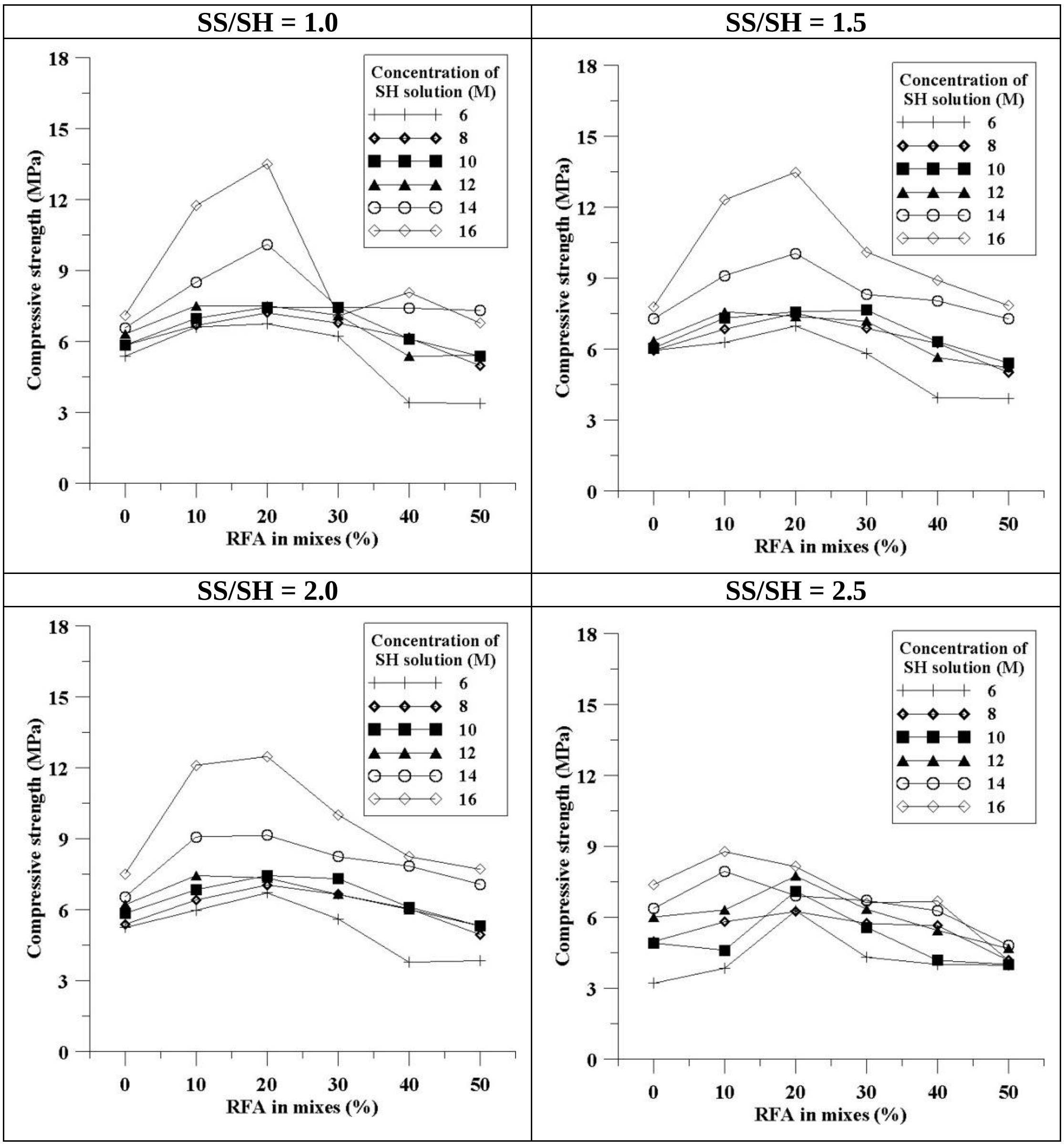


Figure 4.8 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 28 days air curing

From the observations, geopolymer mortar mixes produced with AL consists of SS/SH ratio 1.5, exhibited higher compressive strength at all other parameters. At any concentration of SH solution, SS/SH ratio of 1.5 helps to react with fly ash properly to form three-dimensional silico-aluminate structures, which are mainly responsible for attaining strength. Higher ratio of SS/SH solution in AL leads higher quantity of silicate present in the AL. After getting reacted with fly ash, excess silicate starts to precipitate over fly ash particles, which does not allow furthermore reaction between FA and AL to occur. As a result, three-dimensional silico-aluminate structures formation would not be there. Therefore, strength decrement was observed for the geopolymer mortar mixes produced with AL, when SS/SH ratio was adopted more than 1.5 to prepare the AL.

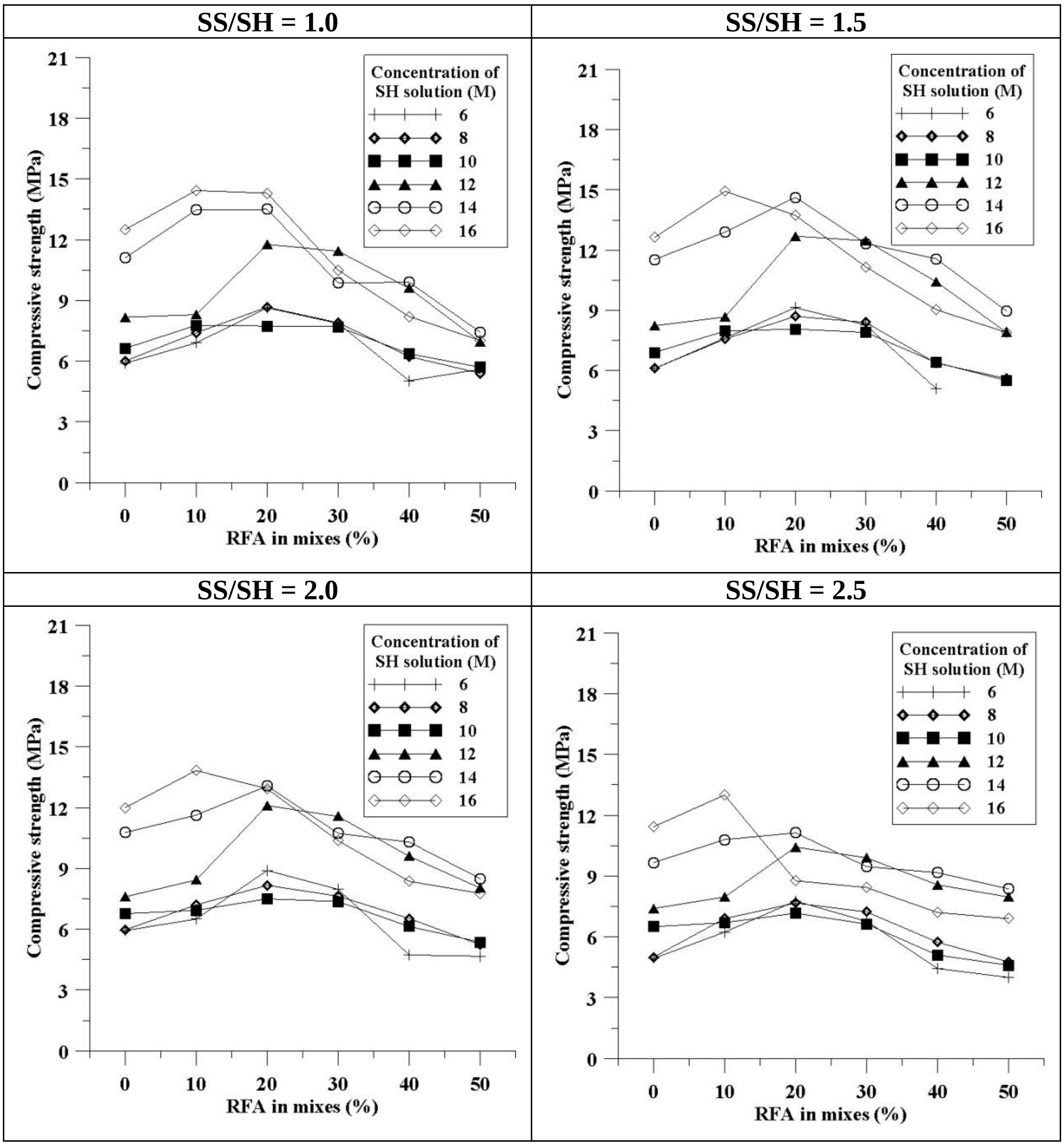


Figure 4.9 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 56 days air curing

4.4.1.3 *Effect of AL/FA ratio*

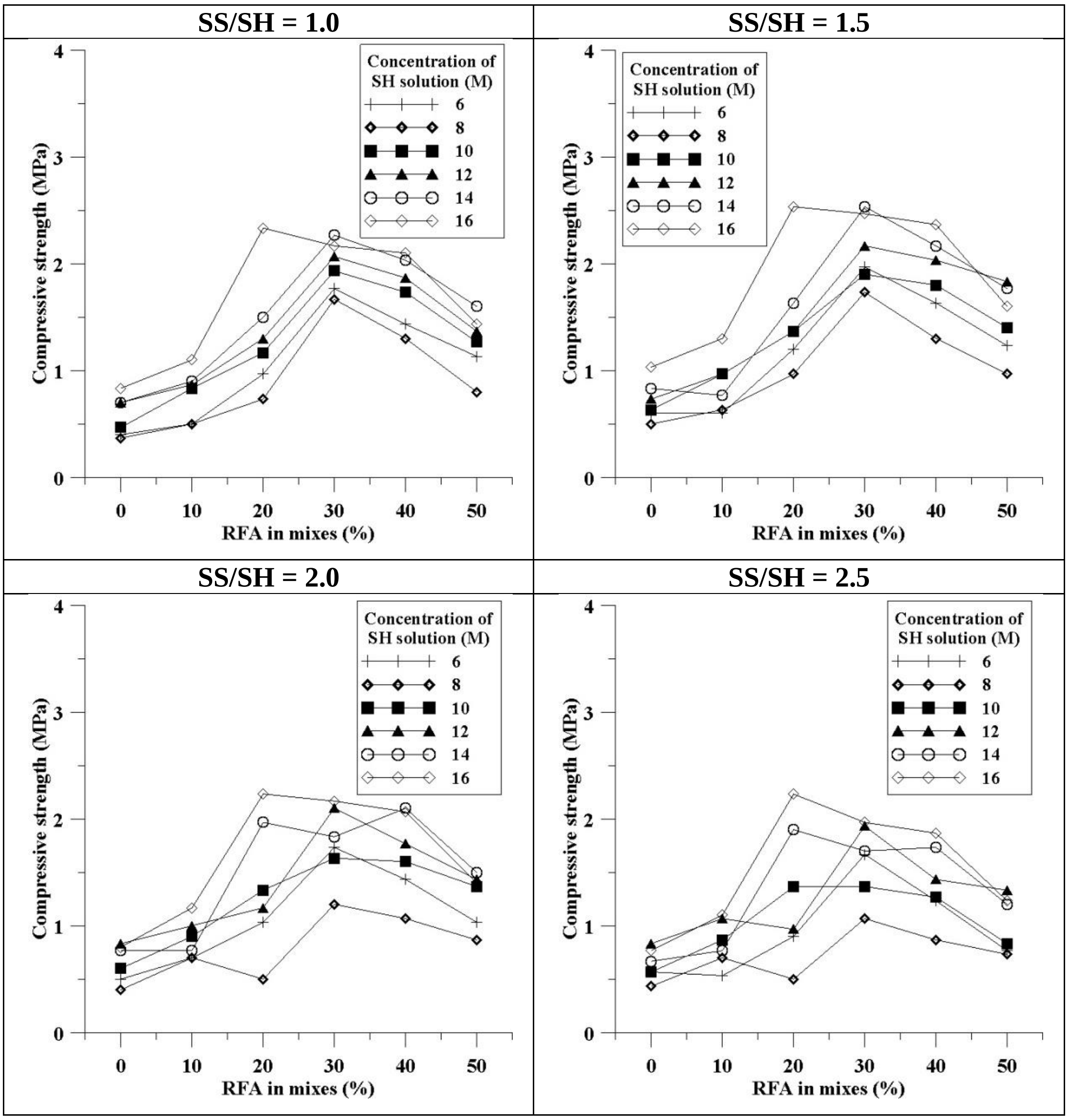


Figure 4.10 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 3 days air curing

Compressive strength of geopolymer is governed by the formation of sodium silico-aluminate gel and poly-condensation to form 3-D network of silico-aluminate structures.When the ratio of AL to FA increased, then the compressive strength of mortar cubes also increased. As total quantity of AL in the mixes is more, which helps to get reacted with FA fully and form the strength attributing silico-aluminate structures. As a result, stable 3-D network of silico-aluminate structures is produced to provide higher strength to the FA based geopolymer mortar mixes. Therefore, geopolymer mortar mixes produced with the consideration of AL/FA = 0.6 showed higher compressive strength than that of AL/FA = 0.4. After 28 days, maximum of 13.5 MPa compressive strength was observed for the mortar mix produced with AL/FA = 0.4 whereas 24.3 MPa of compressive strength was observed as the maximum for the geopolymer mortar mix produced with AL/FA = 0.6. It is clearly seen that 1.8 times higher the strength is achieved with the consideration of AL/FA = 0.6 to prepare mortar mixes.

4.4.1.4 Effect of RFA content

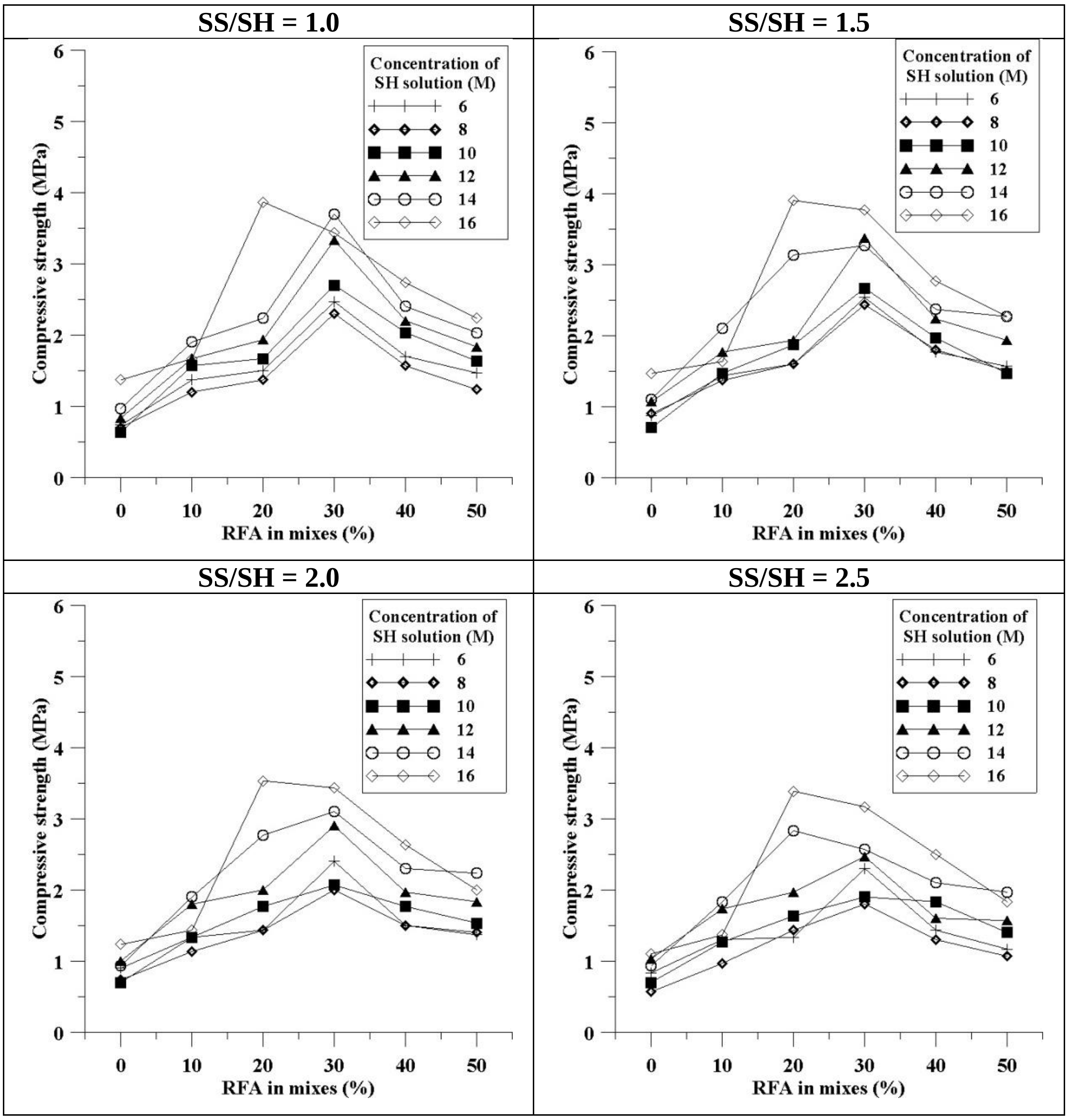


Figure 4.11 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 7 days air curing

It has been observed that compressive strength of mortar cubes after ambient temperature curing was in increasing trend when the replacement level of NFA by RFA was made up to 20% for the mixes generatedwith AL/FA = 0.4. When the replacement level of sand by RFA was beyond 20%, then the strength started decreasing, but most of the mixes exhibited better strength than the control mixes. Higher compressive strength was found for all the mixes produced with 20% RFA as fine aggregate.

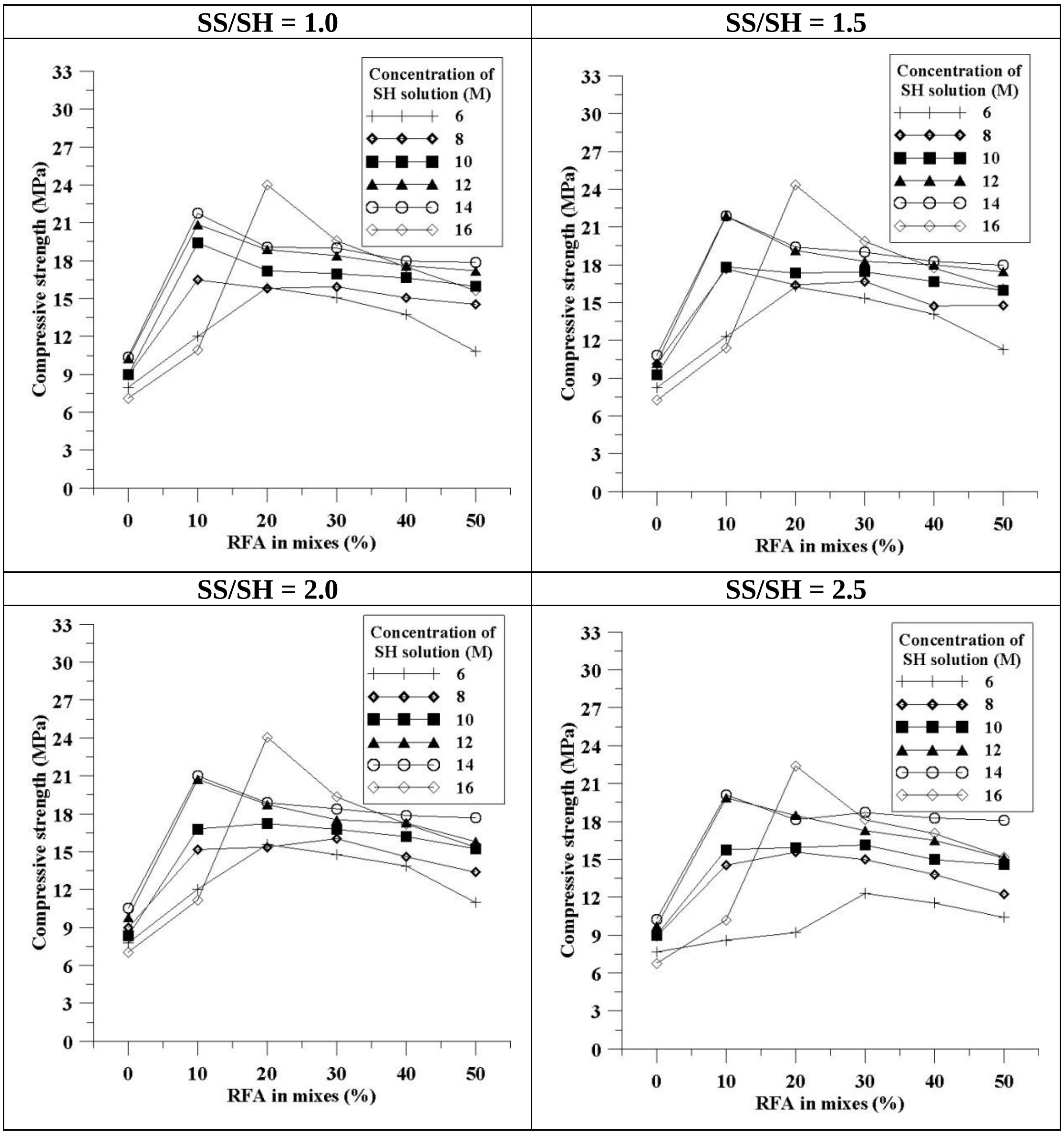


Figure 4.12 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 28 days air curing

For the mortar mixes produced with AL/FA = 0.6, compressive strength of mortar cubes after ambient temperature curing for 3 days and 7 days was in increasing trend when the replacement level of NFA by RFA was made up to 30%. When the replacement level of NFAby RFA was beyond 30%, then the strength started decreasing. But after 28 days and 56 days of air curing, mixes with 10% RFA as fine aggregate showed better strength. It has been also observed that all the mixes exhibited higher compressive strength with respect to the control mixes (mortar mixes with 0% RFA).

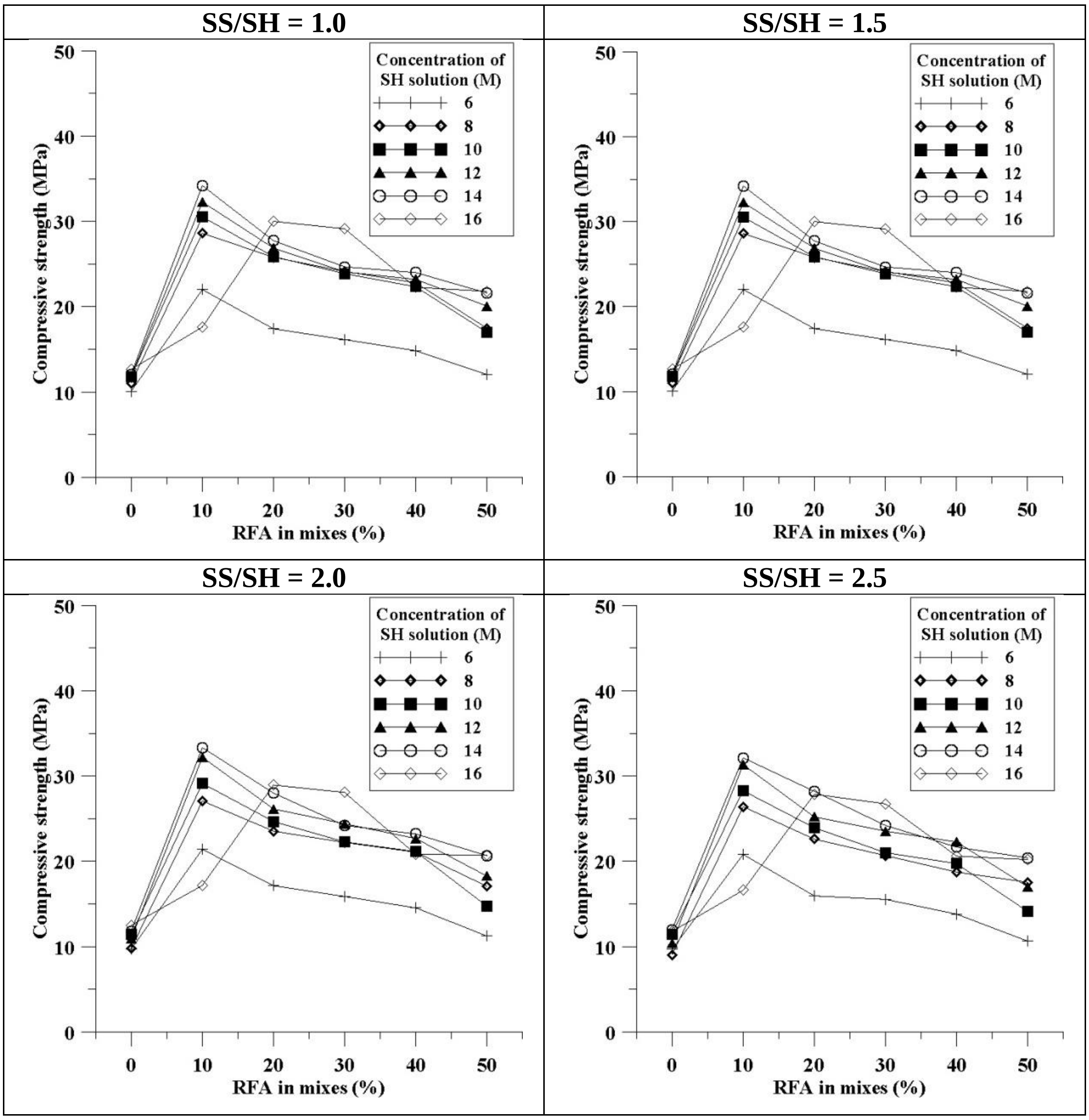


Figure 4.13 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 56 days air curing

Mortar mixes having higher percentages of RFA showed lesser strength because of quality of RFA, higher water absorption capacity of RFA. RFA may absorb water molecules from the alkaline liquid and therefore the geopolymeric reaction might get affected. But most of the mortar mixes showed higher strength than the control mixes (having 0% RFA). It is because mortar mixes with RFA would set well than the mixes having no RFA. In RFA, the hydrated C-S-H gel or un-hydrated cement particles help the mortar mixes to set well.

4.4.2 Water Curing

Figure 4.14 - 4.17 and Figure 4.18 - 4.21 represents the compressive strengthof the water cured geopolymer mortar mixes produced with the consideration of AL/FA = 0.4 and 0.6 respectively. Compressive strength of water cured mortar cubes wasobserved to be lesser than that of air cured. As RFA is presentin mixes, it absorbs water from the curing chamber and this leads to the dilution of the alkaline liquid present in the mixes. Therefore, reaction between alkaline liquid and FA gets disturbed. Thus, formation of the silico-aluminate gel and stabilize silico-aluminate structure as strength attributing factor is also affected.

As per obtained result for water cured samples, most of the mortar mixes produced with AL/FA ratio 0.4 could be categorized as “O” type of mortar according to the guidelines of ASTM C270 (Table 5.1). But few mixes are there of “S” type of mortar. Most of the mortar mixes produced with AL/FA = 0.6 could be categorized as either “N” or “S” type of mortar. But very few mixes are there of “M” type of mortar.

When mortar cube samples were cured under water, then mortar mixes with AL/FA = 0.6 conforms to the grade of either MM3 or MM7.5 as per the Indian Standard (IS: 2250-1981) guidelines and mortar mixes produced with AL/FA = 0.4 conforms to the grade of mortars from MM1.5 to MM5.

4.4.2.1 Effect of the concentration of SH solution

For AL/FA ratio of 0.4, compressive strength of water cured geopolymer mortar cubes was also observed to be increased with the increase of the concentration of SH solution. Highest compressive strength was found for most of the mortar mixes with

16M SH solution. Mortar cubes cured under water showed higher strength for higher concentration of SH solutions in AL.

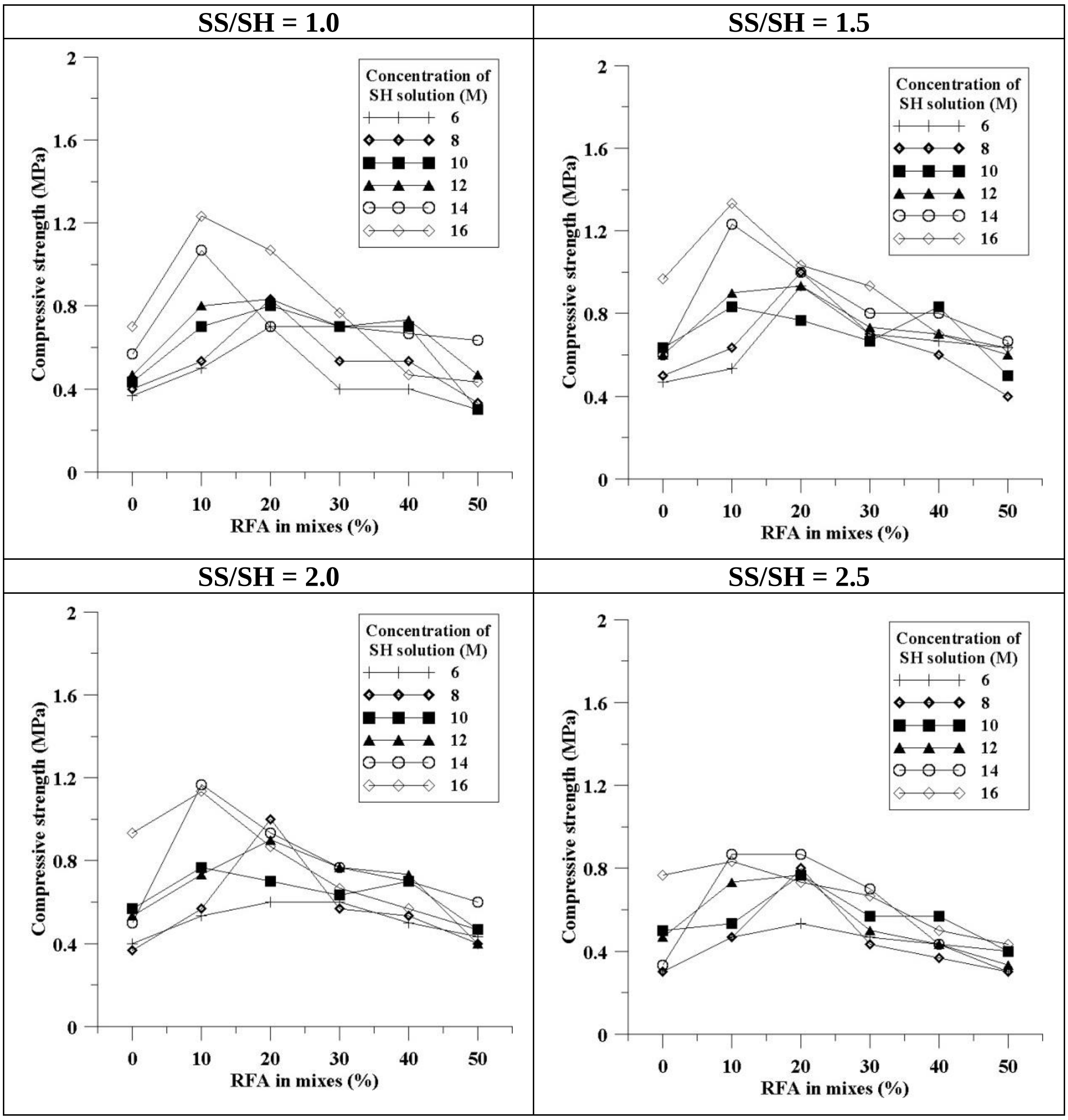


Figure 4.14 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 3 days water curing

For AL/FA ratio of 0.6, compressive strength of cubes was observed to be higher for the mortar mixes produced with higher concentration of SH solution in AL. Mortar mixes with 16M SH solution showed higher compressive strength after 3 days of water curing. But after 7 days, 28 days and 56 days, mortar mixes produced with AL having 12M SH solution exhibited higher compressive strength than that of other mixes.

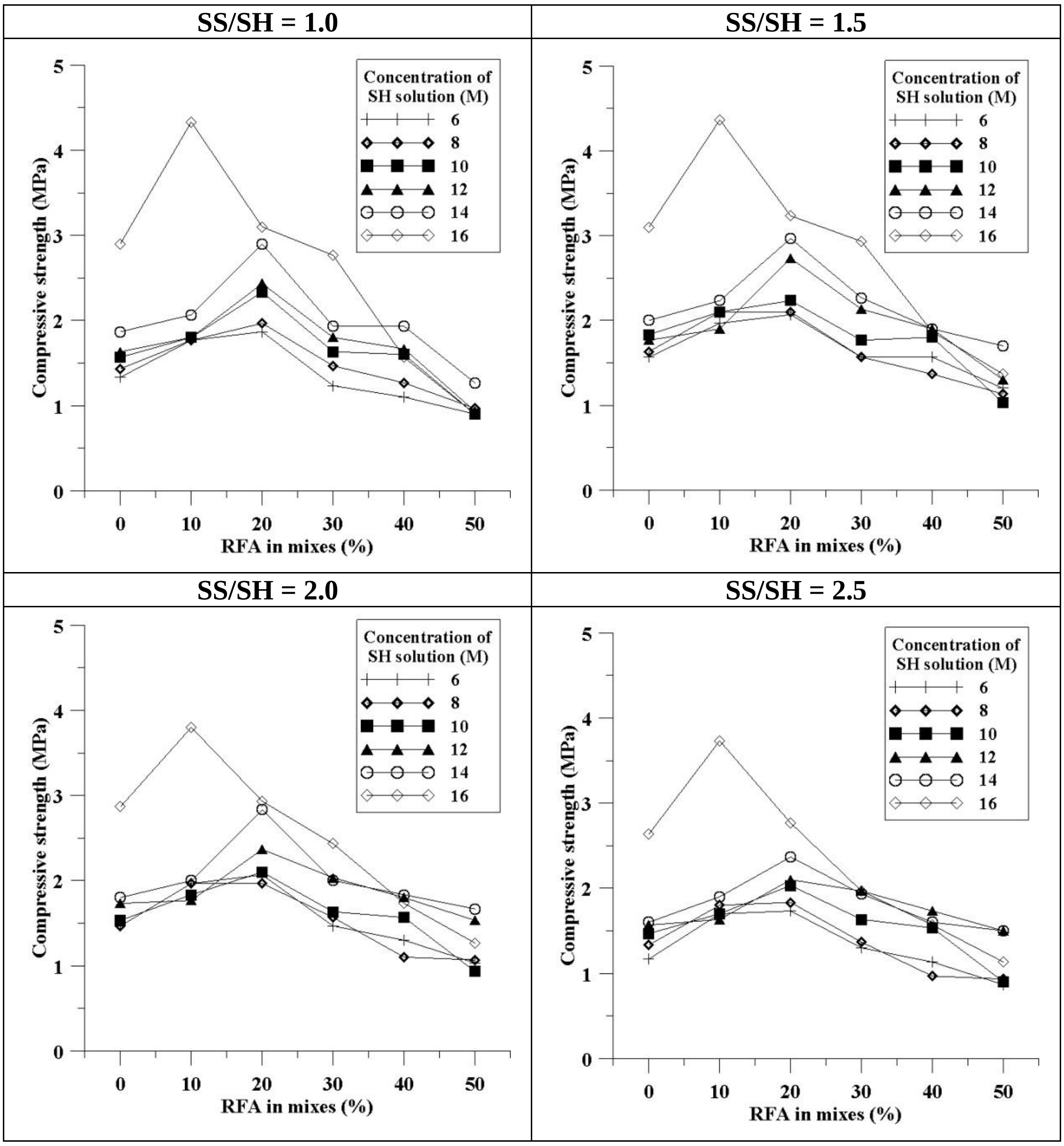
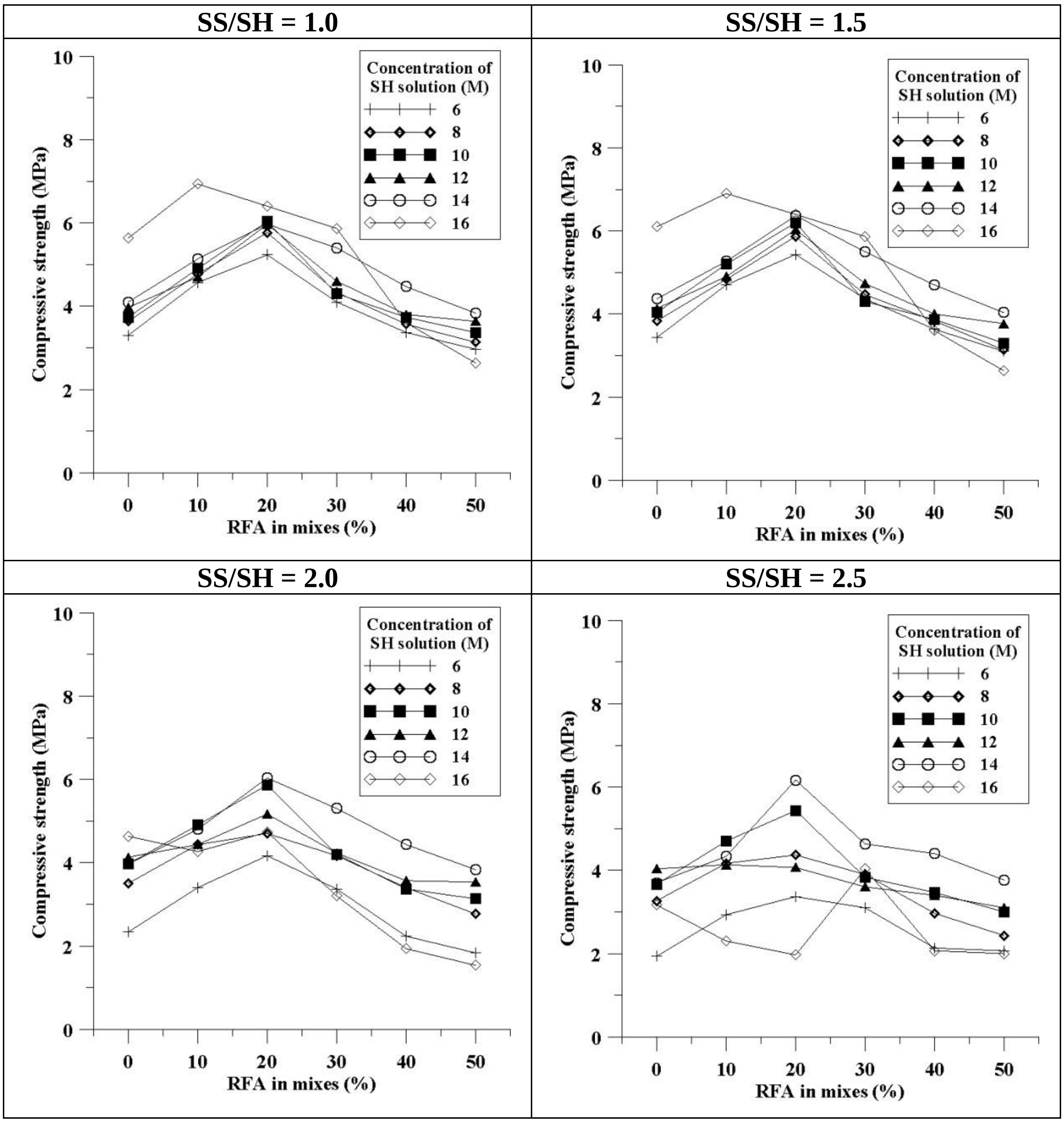


Figure 4.15 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 7 days water curing

4.4.2.2 *Effect of SS/SH ratio*

Geopolymer mortar mixes produced with AL consists of SS/SH ratio of 1.5 exhibited better compressive strength at all other parameters. The variation of compressive strength for all SS/SH ratio at a particular concentration of SH solution in AL was almost similar. SS/SH ratio of 1.5 helps to react with FA properly to form three dimensional silico-aluminate structures, which are mainly responsible for attaining strength at any concentration of SH solution in AL. Higher ratio of SS/SH solution in AL leads higher quantity of silicate present in the AL. After getting reacted with FA, excess silicate starts to precipitate which will not allow furthermore reaction between AL and FA to occur. As a result, three-dimensional silico-aluminate structures formation will not be present. Therefore, strength decrement was observed for the geopolymer mortar mixes produced with AL when SS/SH ratio is adopted more than 1.5 to prepare the AL.

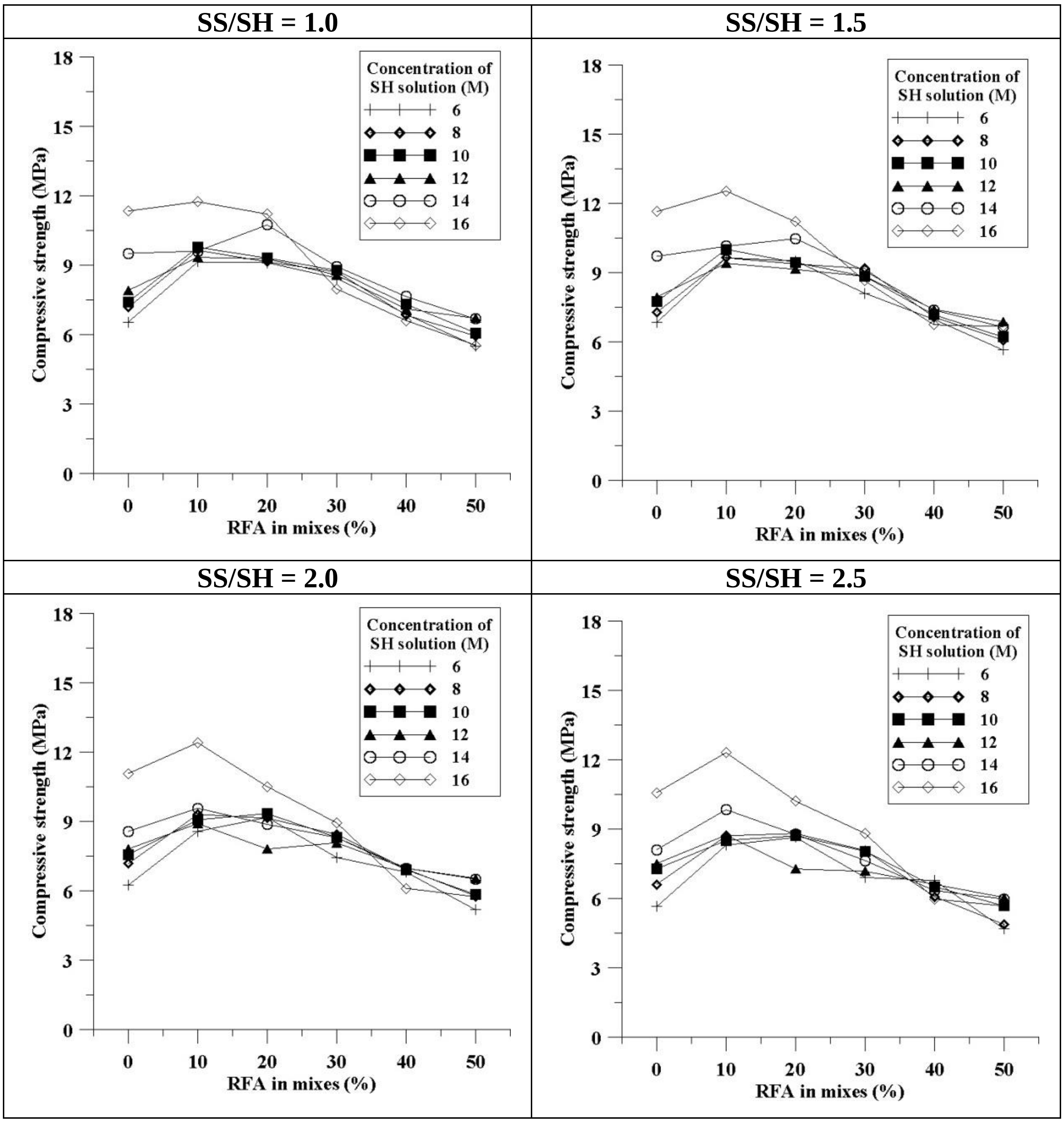

SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.16 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 28 days water curing


SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.17 Compressive strength of geopolymer mortar produced with AL/FA=0.4 after 56 days water curing

4.4.2.3 *Effect of AL/FA ratio*

Formation of silico-aluminate gel and poly-condensation to form 3-D network of silico-aluminate structures help to gain the strength to geopolymer mortar mixes. Compressive strength of mortar cubes increased when the ratio of AL to binder increased. Total quantity of AL in the mixhelps to get reacted with FA and form the strength attributing silico-aluminate structures. As a result, FA based geopolymer mortar mixes produced with AL/FA = 0.6 consists of sufficient amount stable 3-D network of silico-aluminate structures to provide higher strength than that of the mortar mixes produced with AL/FA = 0.4. But mortar mixes with AL/FA = 0.6, exhibited very less water absorption capacity and therefore, the reaction between AL and FA in these mixes was less affected. Therefore, these mixes exhibited higher strength than mixes produced with AL/FA = 0.4. After 28 days of water curing, highest strength of 6.9 MPa was observed for the mixes produced with AL/FA = 0.4 whereas 19.9 MPa was observed for the mixes produced with AL/FA = 0.6.

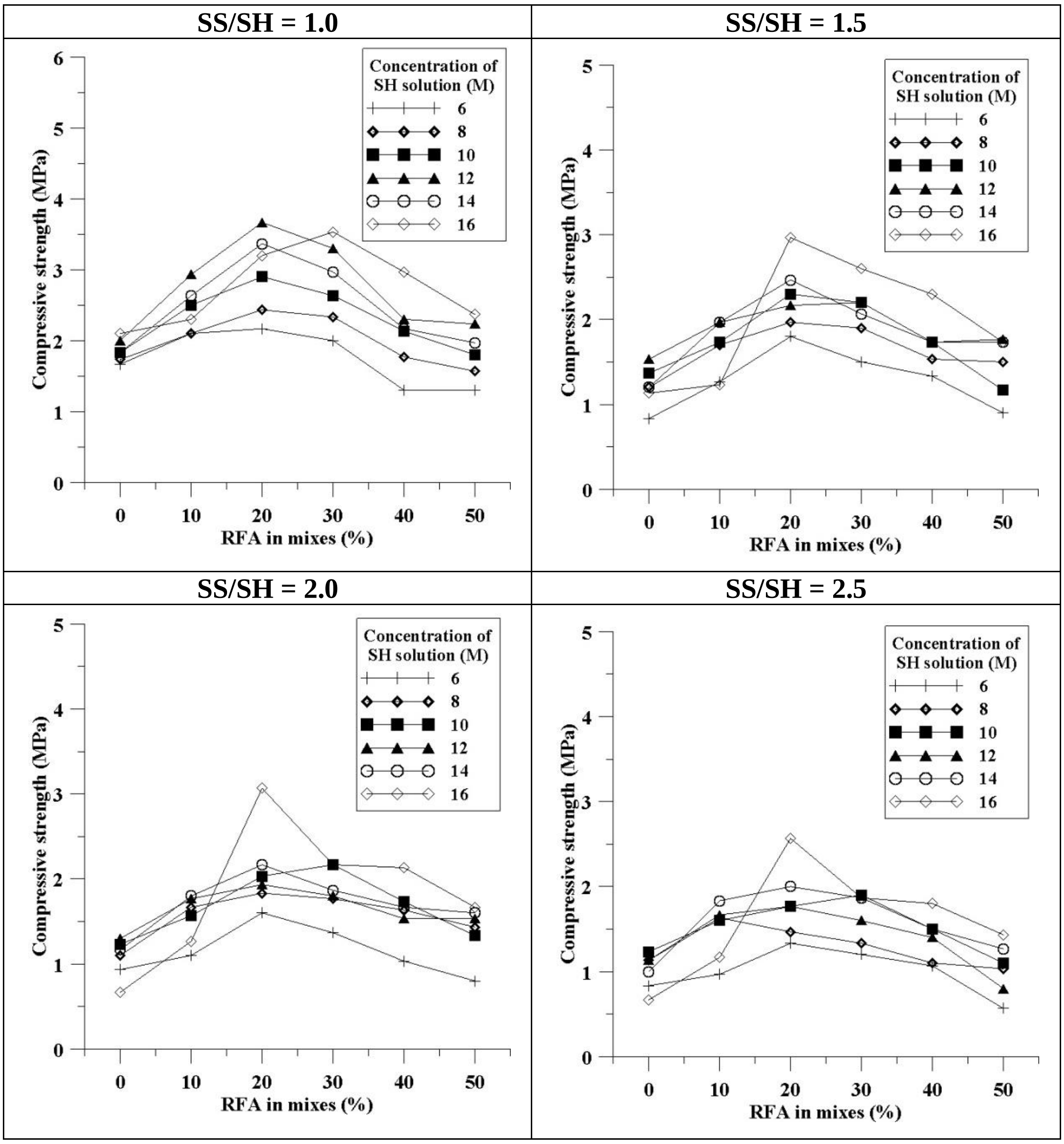


Figure 4.18 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 3 days water curing

4.4.2.4 *Effect of RFA content*

For AL/FA = 0.4, highest compressive strength of mortar cubes cured under normal water was found for the mixes with 10% RFA content. But mortar mixes with 20% RFA content showed consistent performance in strength for all the different mixes. Here also most of the mixes especially upto 30% RFA content performed better than the control mixes.

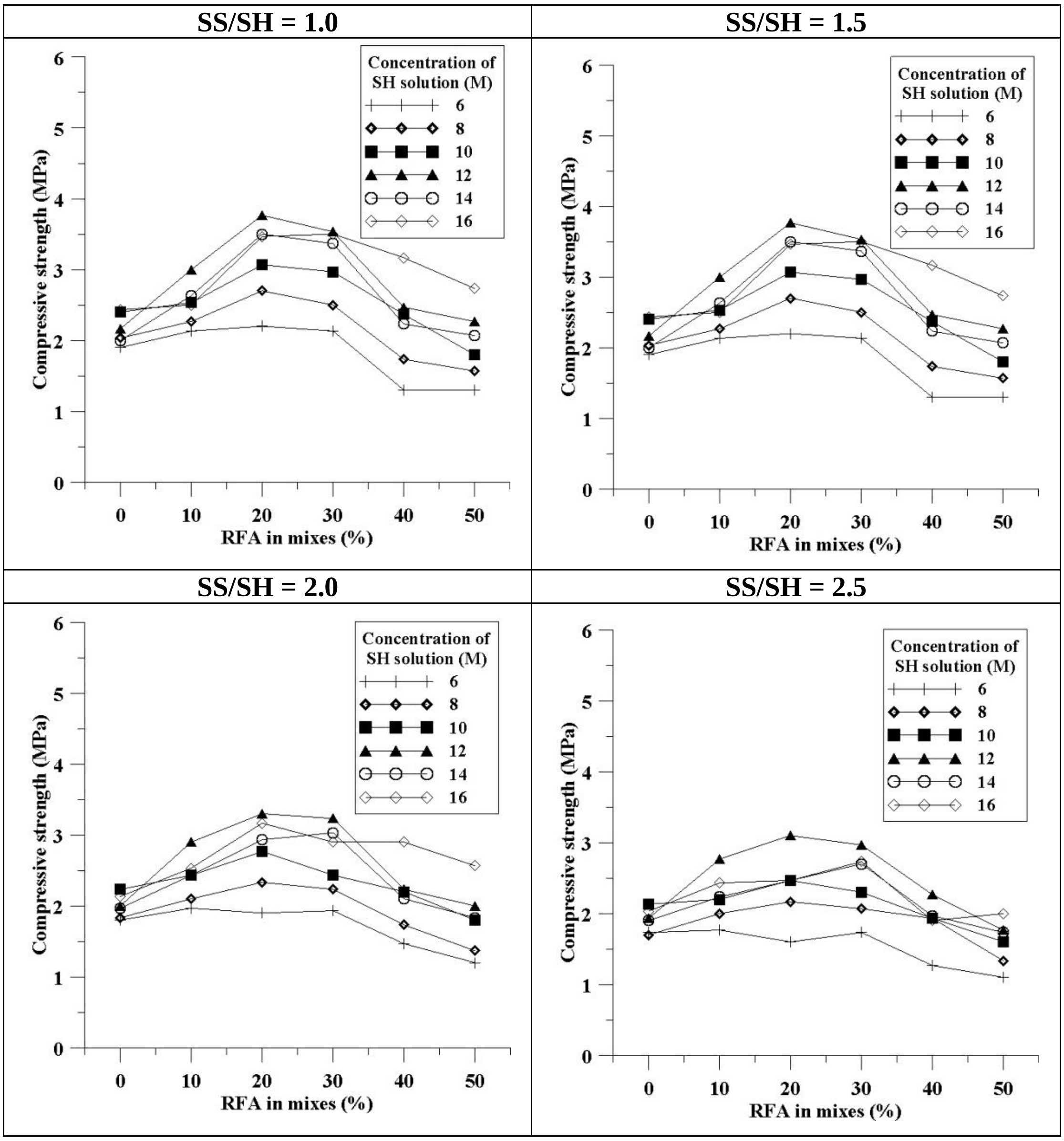


Figure 4.19 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 7 days water curing

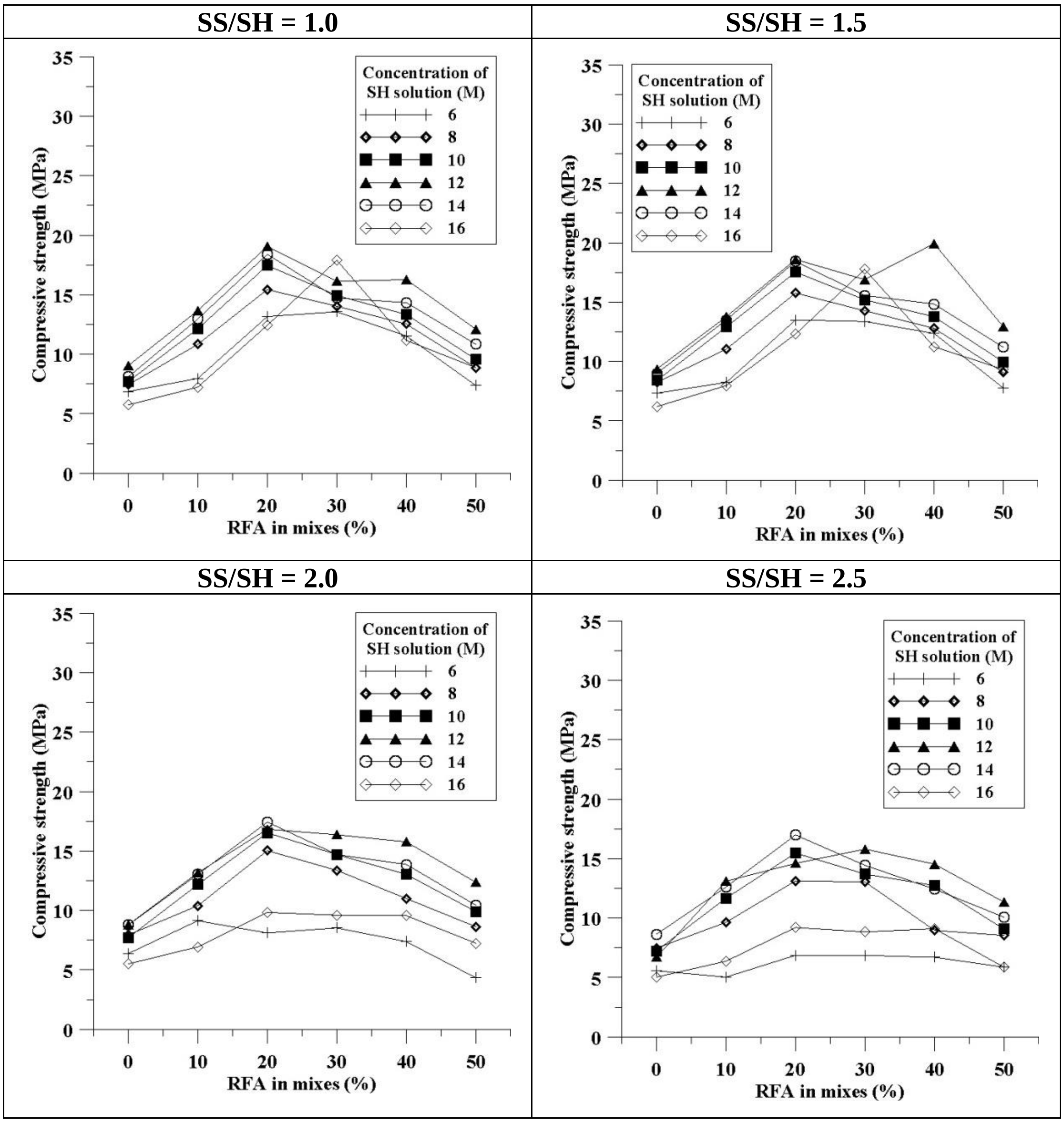


Figure 4.20 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 28 days water curing

It has been observed that FA based geopolymer mortar mixes having 20% RFA exhibited higher compressive strength for water cured mortar cubes. Most of the mortar mixes showed higher compressive strength than the control mixes for the AL/FA ratio of0.6. When mortar cubes were cured under water, mortar mixes having higher percentage of RFA showed higher water absorption especially, when the quantity of AL in the mixes was less. RFA present in the mix absorbs the water from water curing chamber to fulfill their water absorption capacity. This occurrence leads to disturbing the AL present in the entire mixes. Quality of RFA, shape, and size of

RFA etc. also affects the compressive strength of the mixes. Saha and Rajasekaran (2016), Apoorva et al. (2016), Mamery et al. (2013), Neno et al. (2014), Evangelista and de Brito (2007), Kou and Poon (2009) also identified higher water absorption capacity, porous nature, lower density, etc of recycled aggregate as the responsible reasons for the detrimental effect on the properties of concrete/mortar mixes having recycled aggregate.

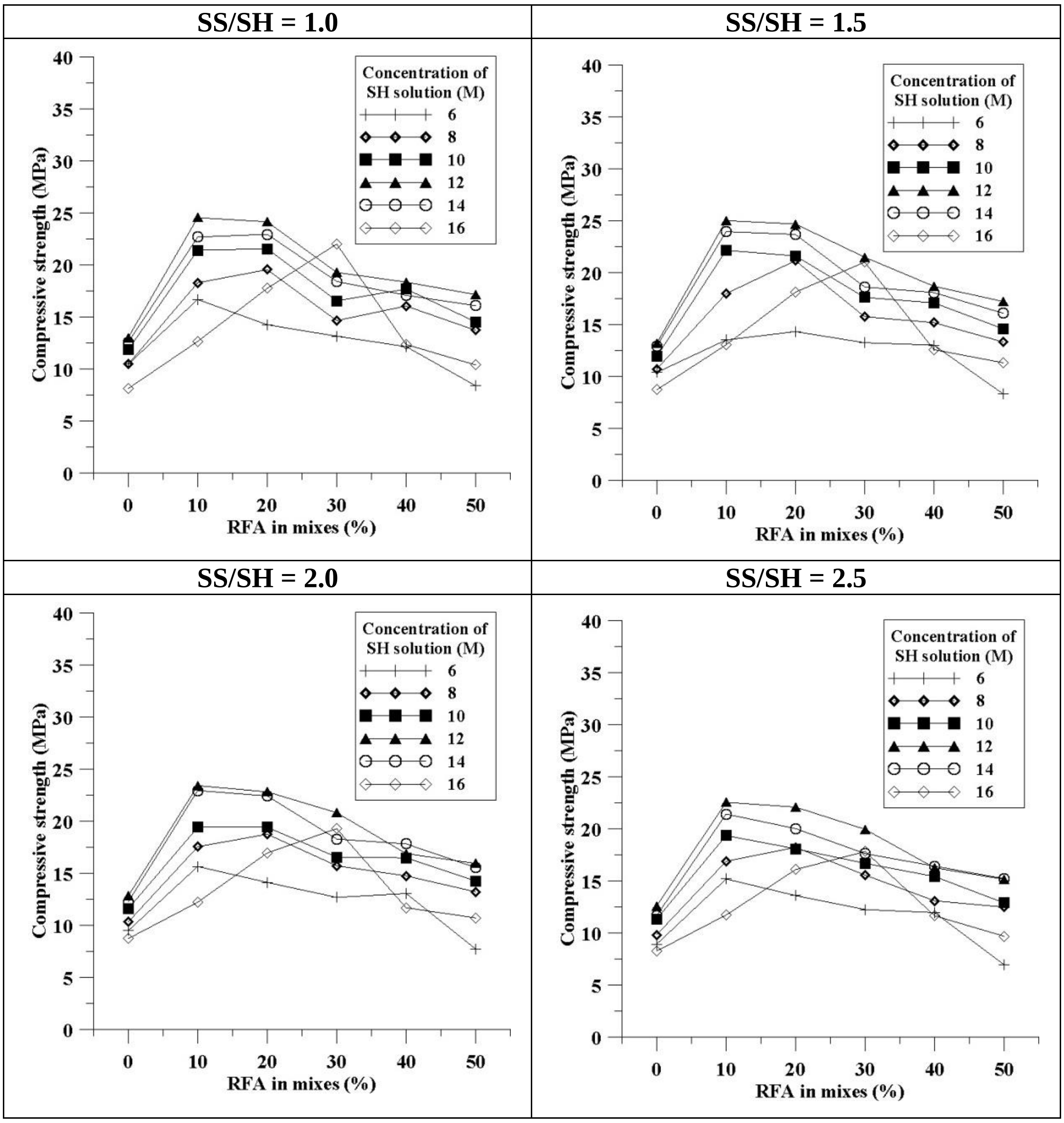


Figure 4.21 Compressive strength of geopolymer mortar produced with AL/FA=0.6 after 56 days water curing

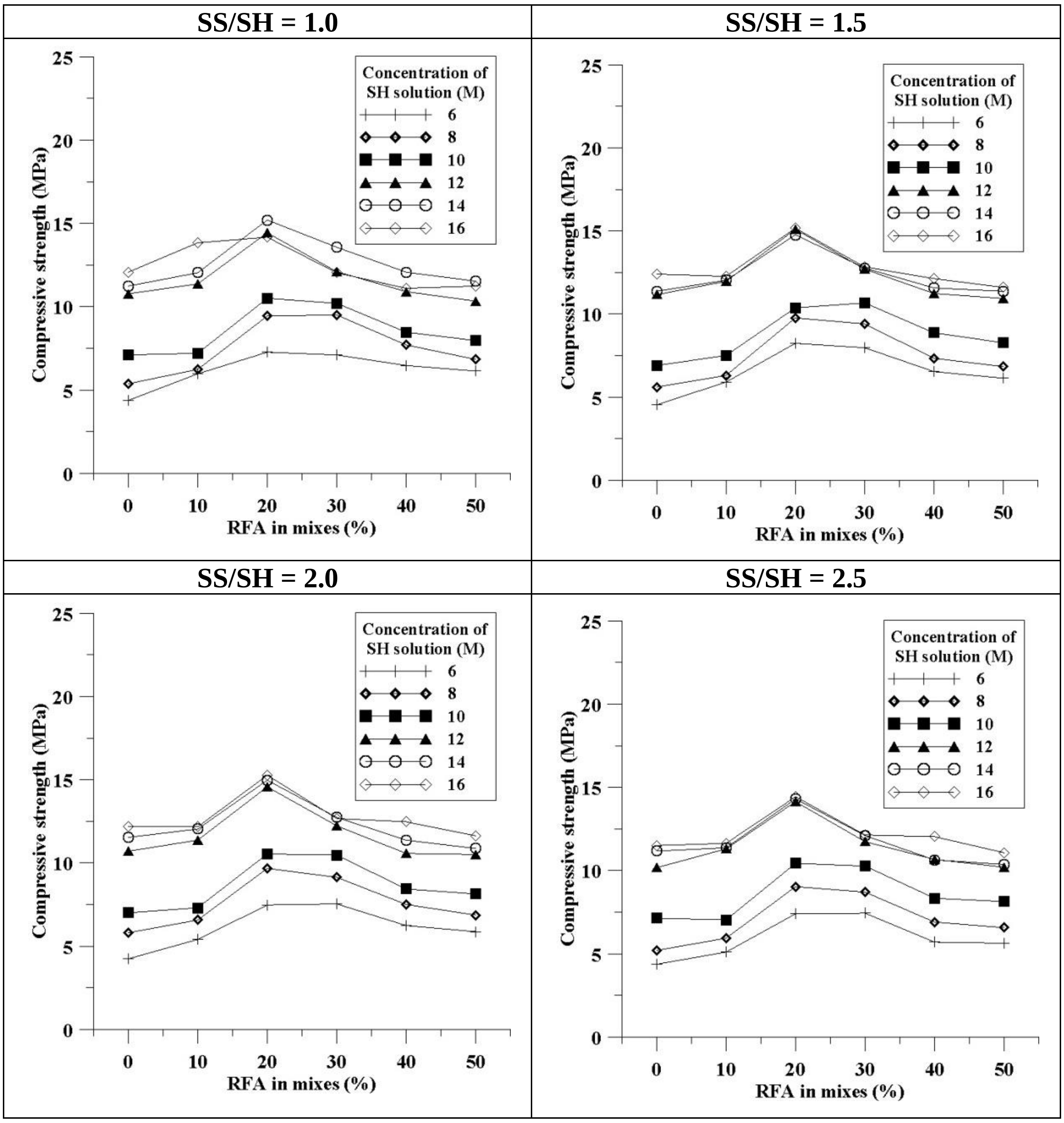
4.4.3 Heat Curing

Compressive strength of geopolymer mortar cubes cured at 80°C for 24 hours showed better than the mortar cubes cured at ambient temperature and under water. Geopolymeric reaction (reaction between FA and AL) becomes faster and effective to form the strength attributing structures for achieving higher strength. Variations of compressive strength of the geopolymer mortar mixes produced with the consideration of AL/FA = 0.4 and 0.6 respectively and cured at 80°C for 24 hours are represented by Figure 4.22 - 4.25 and Figure 4.26 - 4.29. According to ASTM C270, mortar mixes have been categorized into different categories based on compressive strength and mentioned suitable activities to use the different types of mortar effectively. As per obtained result of compressive strength at 28 days for heat cured samples, most of the mortar mixes produced with AL/FA ratio of 0.4 can be categorized as either "N" or "S" type of mortar. Most of the mortar mixes produced with AL/FA ratio of 0.6 can be categorized as either "M" or "N" type of mortar. But very few mixes are there of "S type of mortar.

According to IS: 2250-1981, it has been observed that heat cured mortar cube samples are conforming to the category of MM5 to MM7.5 for the mixes with both AL/FA ratios.

4.4.3.1 Effect of the concentration of SH solution

Compressive strength of FA based geopolymer mortar mixes increased with the higher concentration of SH solution present in AL. Higher concentration of SH solution in the AL helps to react with FA properly and produce the silico-aluminate structures, which is mainly responsible for gaining strength in geopolymeric binders. For AL/FA = 0.4, it is clearly seen two different zones of compressive strength from the graphical representations. Mortar mixes produced with AL having lower concentration of SH solution (6M, 8M,and 10M) exhibited lower compressive strength than the mortar mixes produced with AL having higher concentration of SH solution (12M, 14M and 16M).


SS/SH = 1.0

SS/SH = 1.5

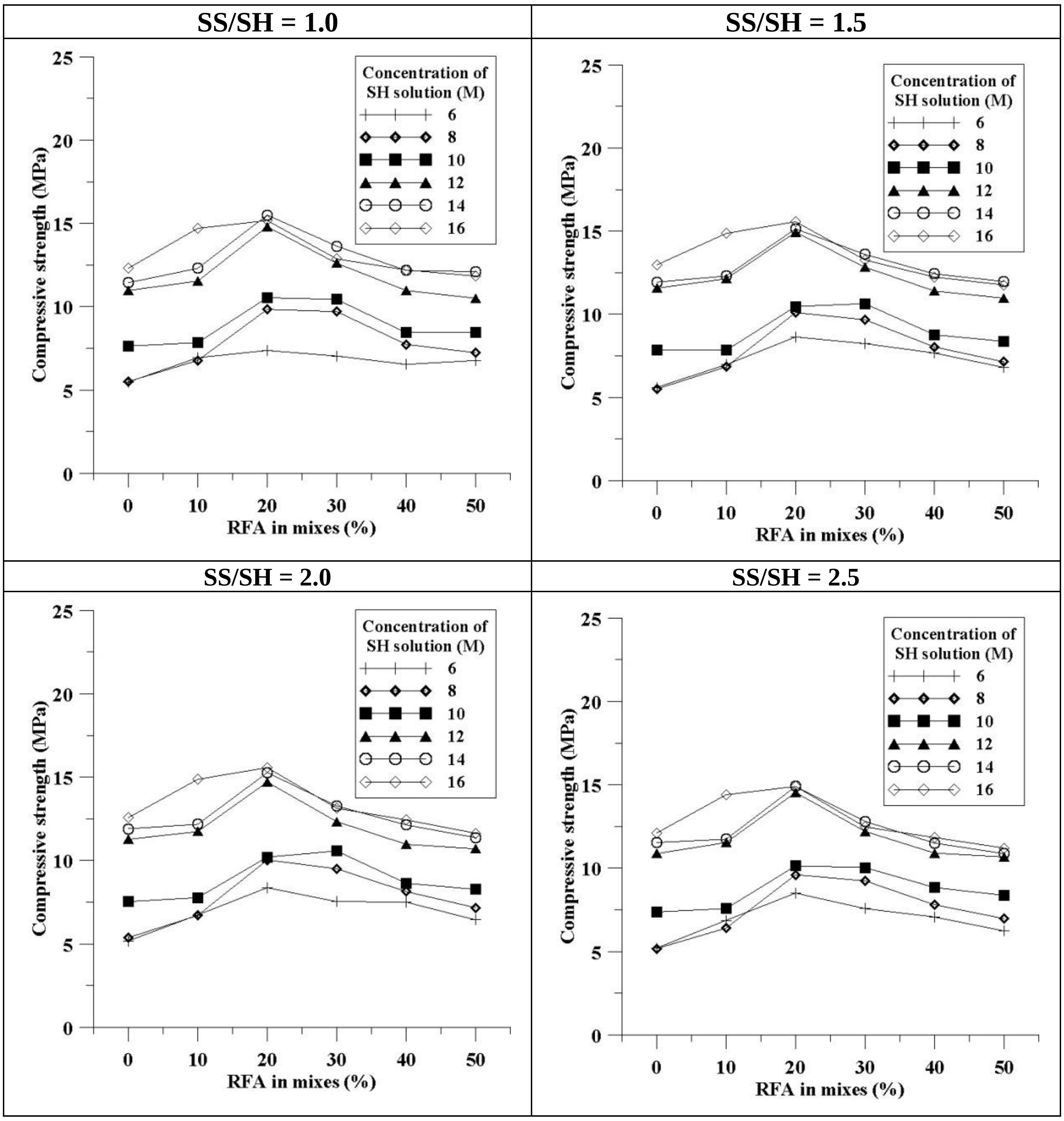
SS/SH = 2.0

SS/SH = 2.5

Figure 4.22 Compressive Strength of geopolymer mortar mixes for AL/FA=0.4 after 3 days (Heat Curing Duration = 24 hours)

4.4.3.2 Effect of SS/SH ratio

All the geopolymer mortar mixes with different ratio of SS/SH solution showed a similar trend of the variation of compressive strength after 3 days, 7 days, 28 days and 56 days. Comparatively, geopolymer mortar mixes produced with AL having ratio of SS/SH solution equal to 1.5 showed higher compressive strength at all other parameters. At any concentration of SH solution, SS/SH ratio of 1.5 helps to react with fly ash properly to form three dimensional silico-aluminate structures, which are mainly responsible for attaining strength. Higher ratio of SS/SH solution in AL leads higher quantity of silicate present in the AL. After getting reacted with FA, excess silicate starts to precipitate which will not allow furthermore reaction to occur. As a result, three-dimensional strength attributing structures formation will be affected. Therefore, strength decrement was observed for the geopolymer mortar mixes produced with AL when SS/SH ratio was adopted more than 1.5 to prepare the AL.



SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.23 Compressive Strength of geopolymer mortar mixes for AL/FA=0.4 after 7 days (Heat Curing Duration = 24 hours)

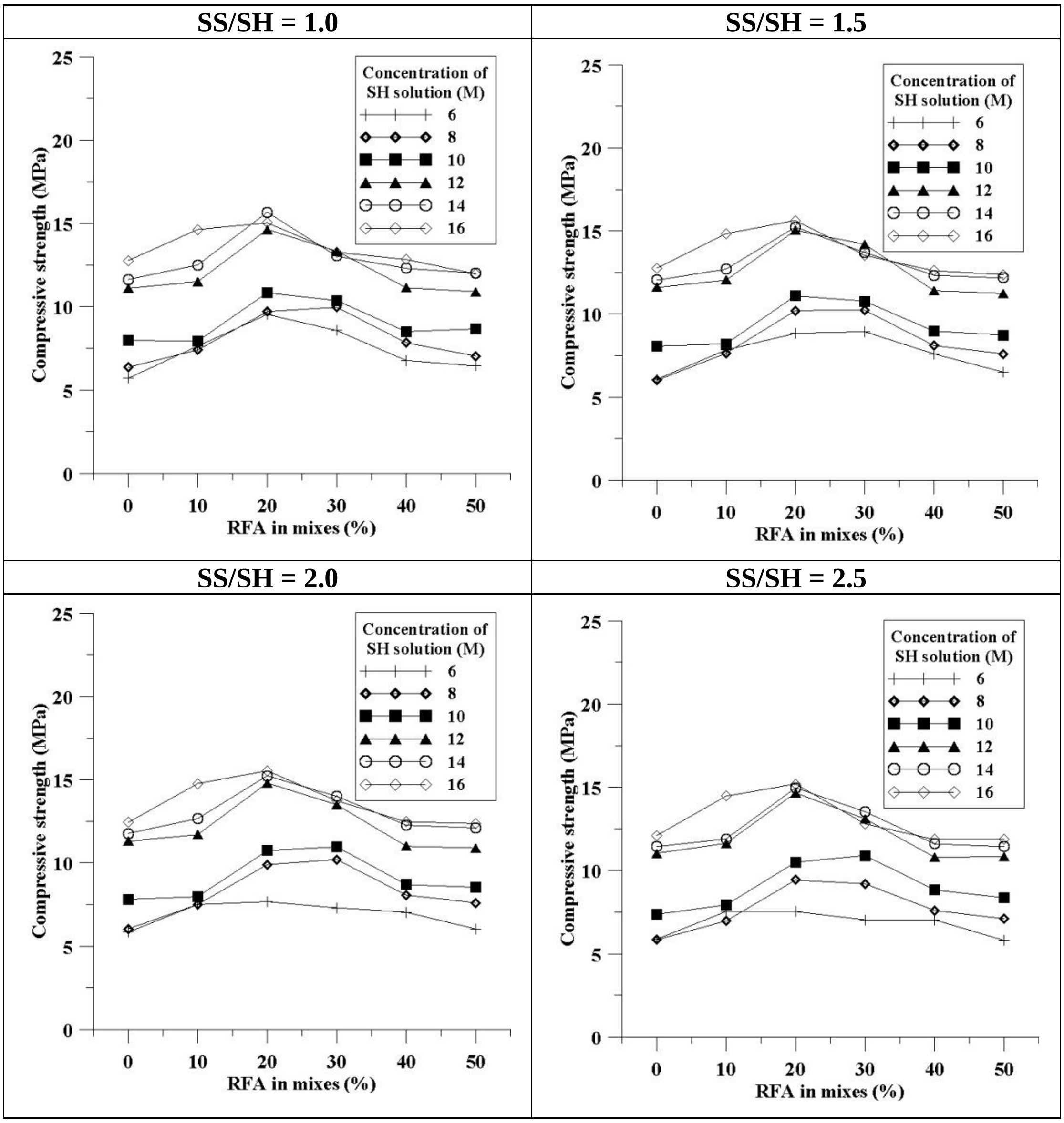
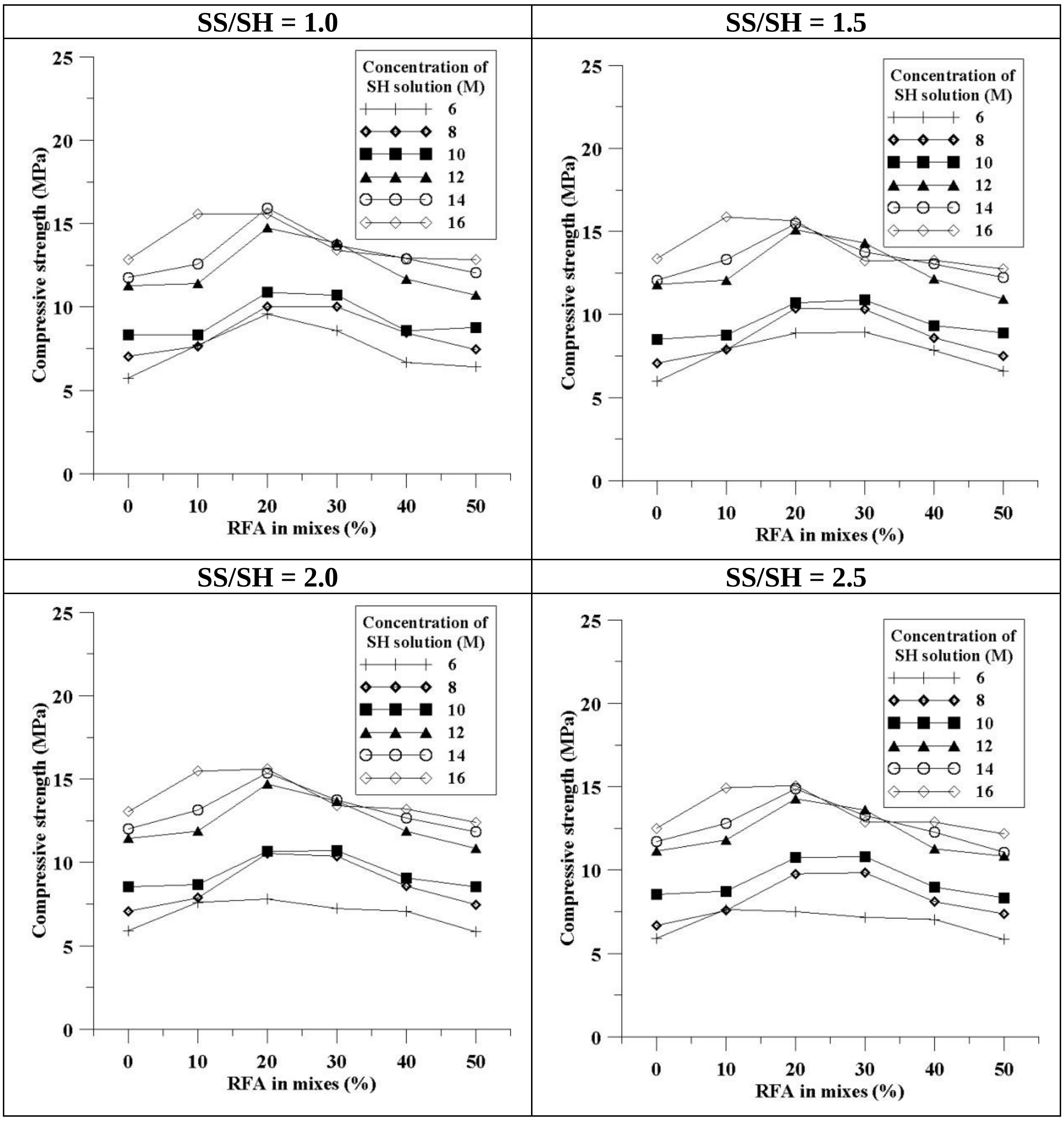


Figure 4.24 Compressive Strength of geopolymer mortar mixes for AL/FA=0.4 after 28 days (Heat Curing Duration = 24 hours)

4.4.3.3 Effect of AL/FA ratio

More compressive strength was observed for the geopolymer mortar mixes produced with higher AL/FA ratio. Lower AL/FA ratio produced harsh mixes and the quantity of AL in the mixes was not enough to get react properly with FA to construct the main responsible silico-aluminate structures to gain strength. FA based geopolymer mortar mixes with AL/FA ratio greater than 0.6 produces more workable mixes but it takes more time to set. After 28 days, maximum of 31.9 MPa compressive strength was observed for the mixes produced with AL/FA = 0.6, whereas mixes produced with AL/FA = 0.4 exhibited maximum of 15.7 MPa compressive strength.


SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.25 Compressive Strength of geopolymer mortar mixes for AL/FA=0.4 after 56 days (Heat Curing Duration = 24 hours)

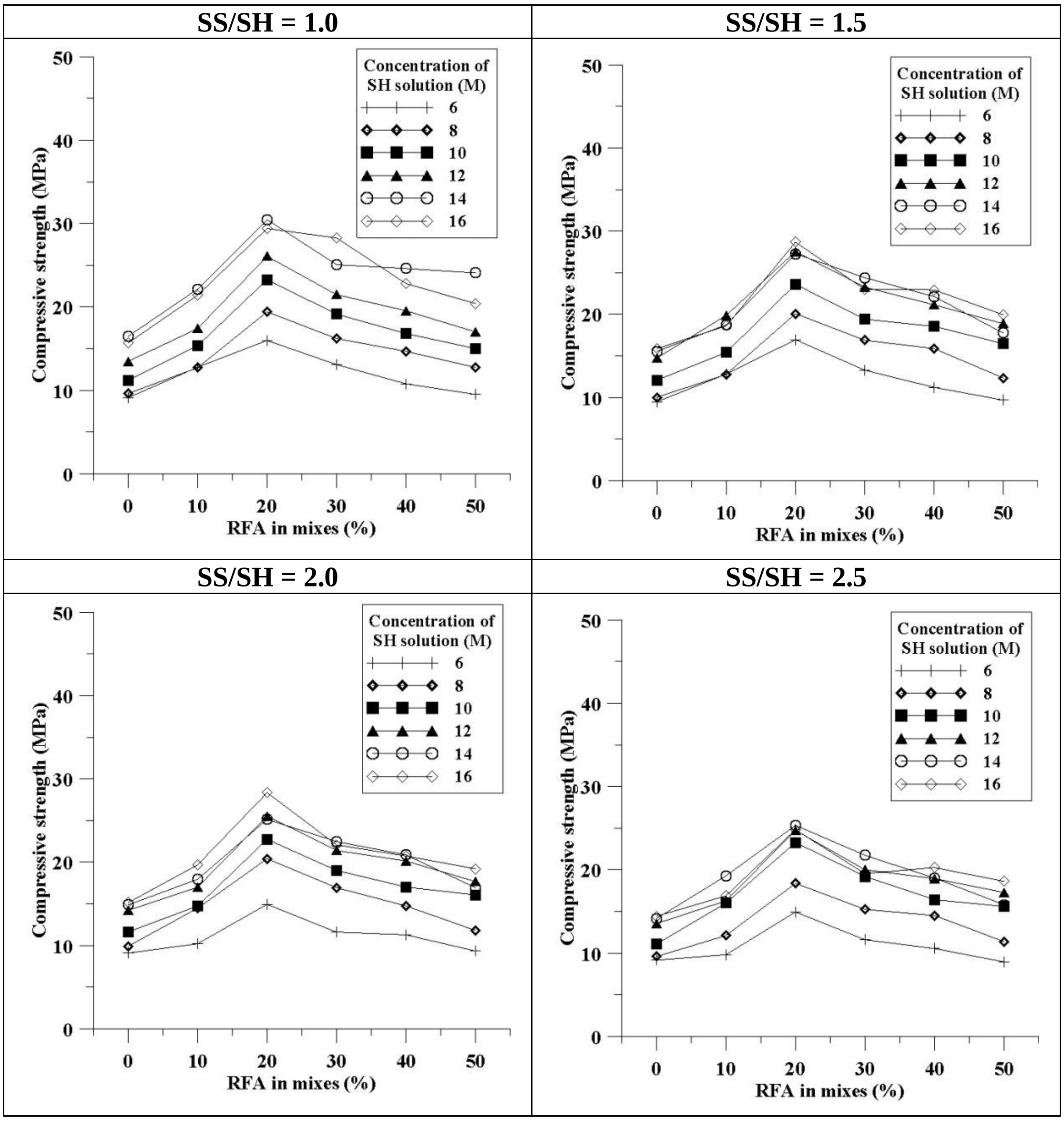


Figure 4.26 Compressive Strength of geopolymer mortar mixes for AL/FA=0.6 after 3 days (Heat Curing Duration = 24 hours)

4.4.3.4 Effect of RFA content

Most of the FA based geopolymer mortar mixes exhibited increasing trend for the compressive strength till the RFA content upto20% in the mixes. Mortar mixes produced with both AL/FA ratio, show almost similar trend of compressive strength after 3days, 7 days, 28 days and 56 days. Quality of RFA has major role in the mixes to attain adequate strength. In the present study, water absorption capacity of RFA was observed to be significantly higher than the natural fine aggregate. This property of RFA provides important contribution to the reduction of the strength when the percentage of RFA was more in the mixes. It absorbs water molecules from the AL and disturbs the chemical equilibrium of AL so that the reaction between FA and AL get affected to create the strength attributing products. As a result, strength decrement was observed with higher RFA content in mixes.

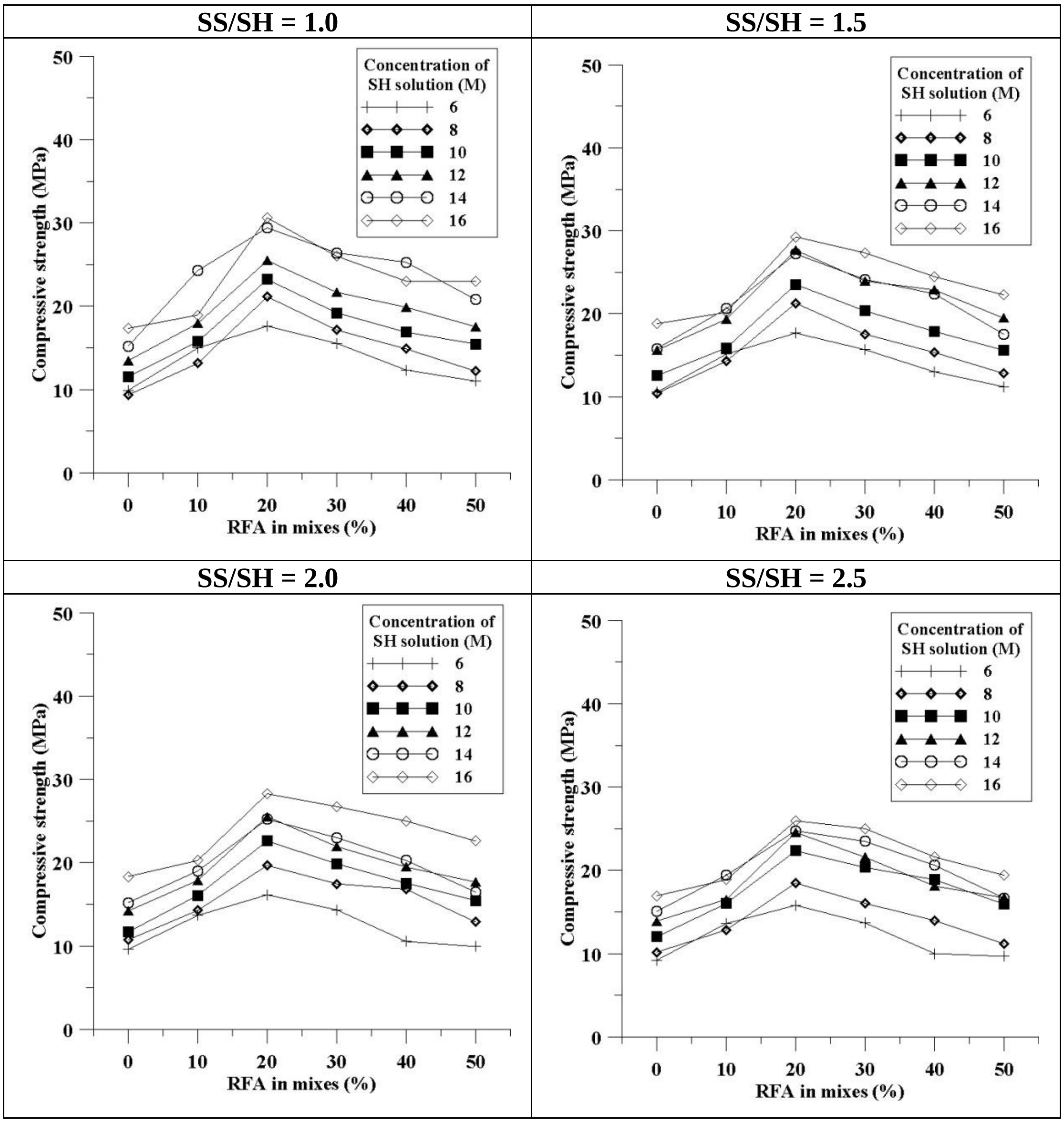
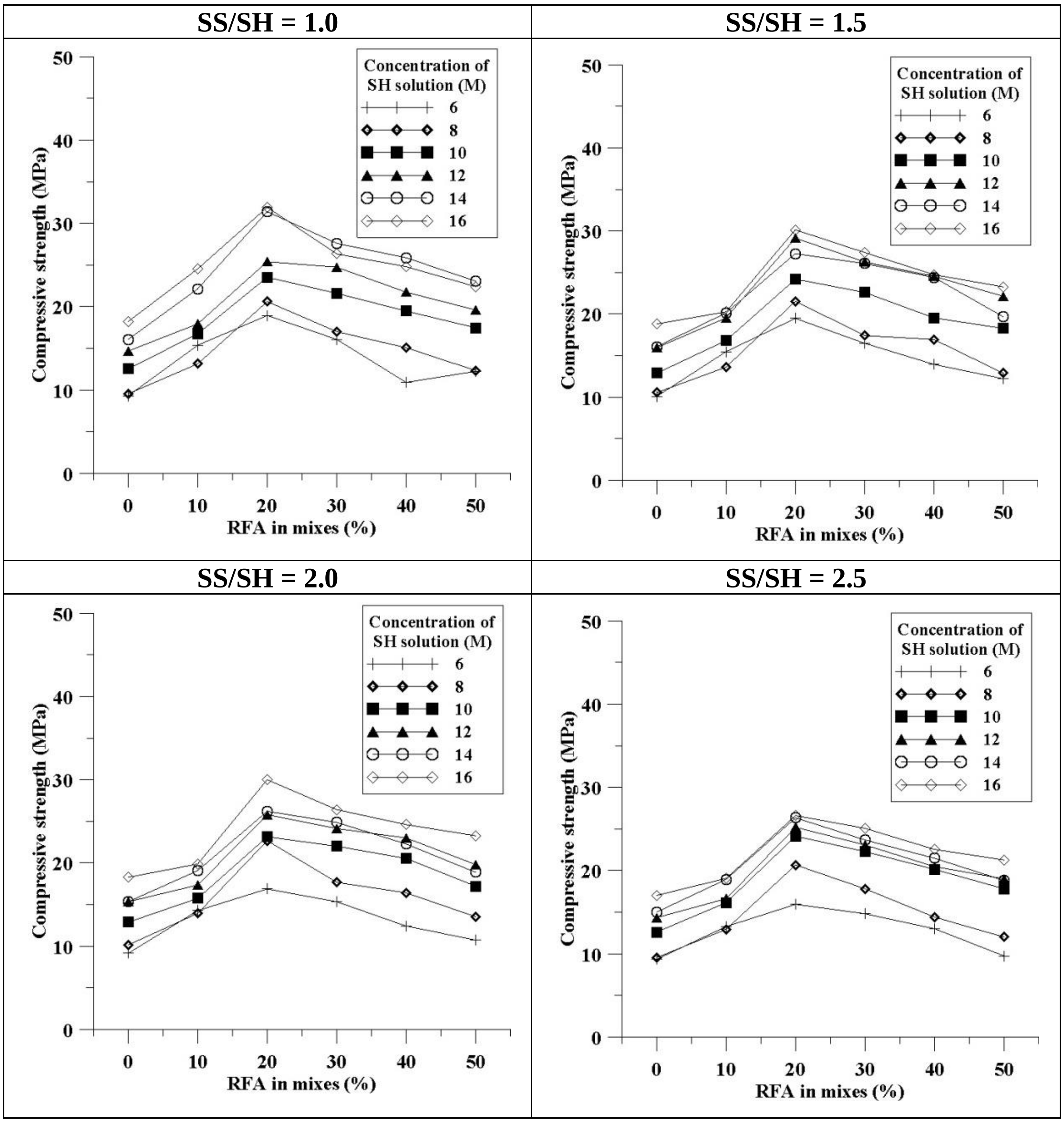


Figure 4.27 Compressive Strength of geopolymer mortar mixes for AL/FA=0.6 after 7 days (Heat Curing Duration = 24 hours)


SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.28 Compressive Strength of geopolymer mortar mixes for AL/FA=0.6 after 28 days (Heat Curing Duration = 24 hours)

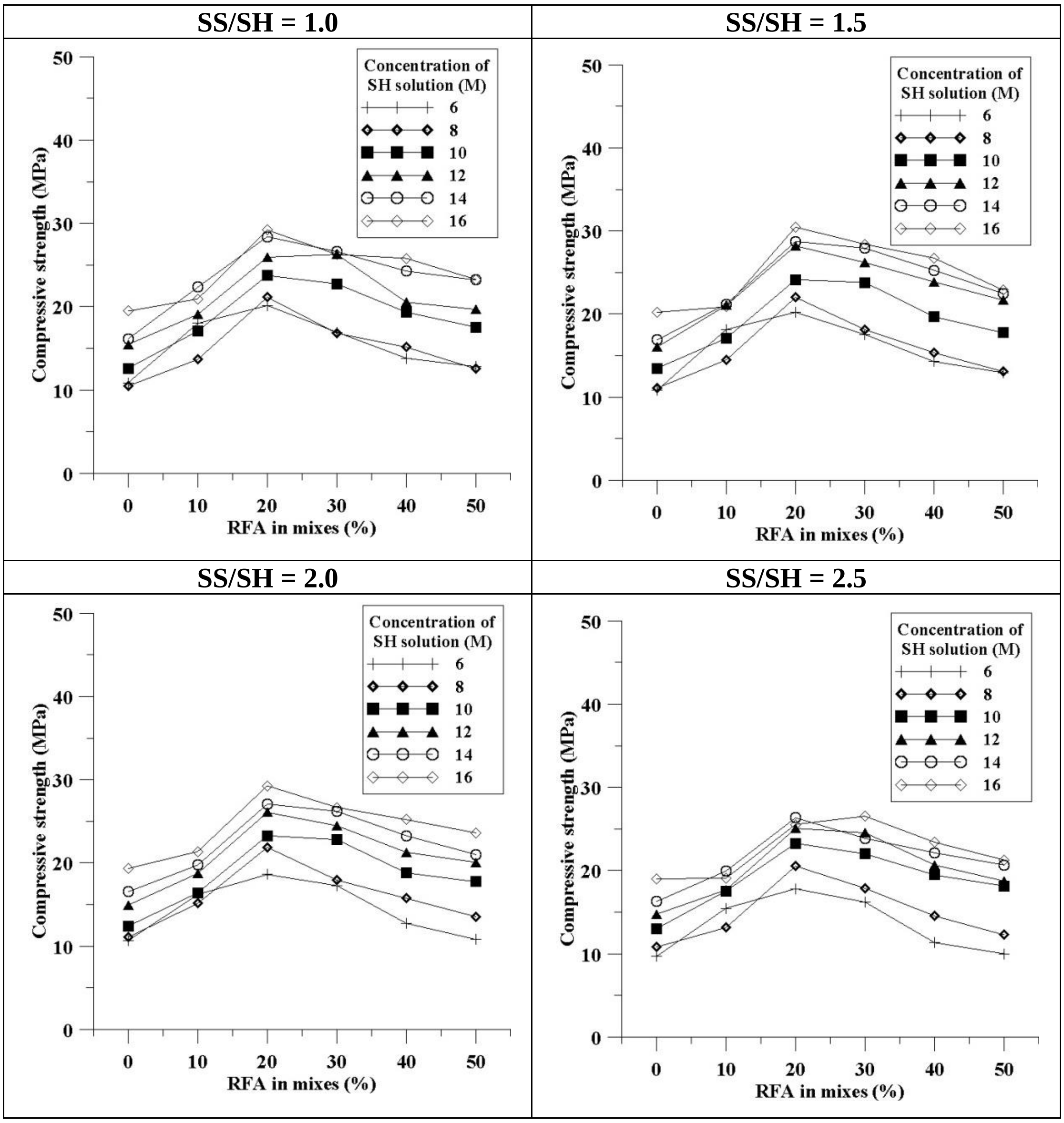
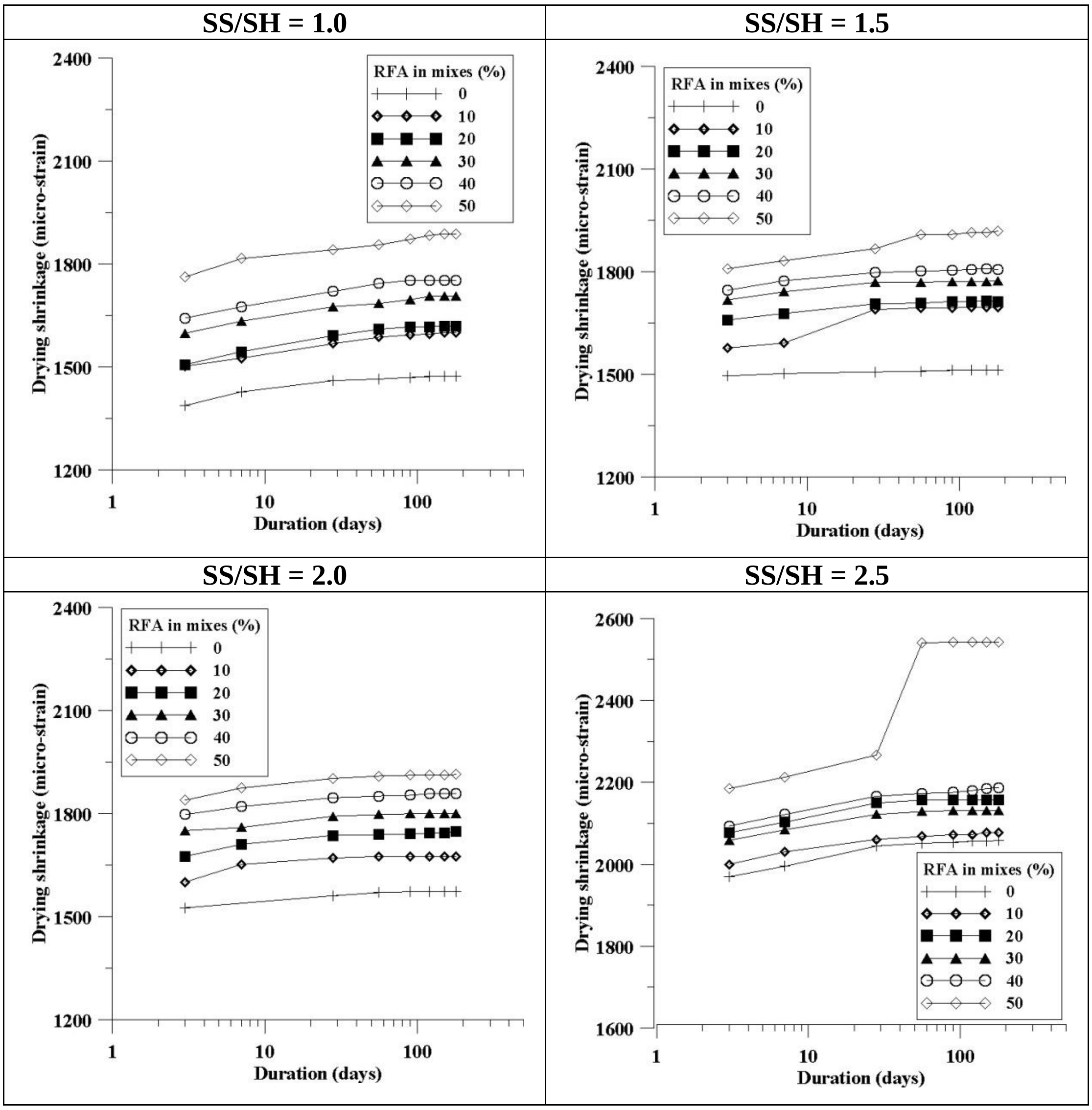


Figure 4.29 Compressive Strength of geopolymer mortar mixes for AL/FA=0.6 after 56 days (Heat Curing Duration = 24 hours)

4.5 DRYING SHRINKAGE

Drying shrinkage of any type of mortar mixes is nothing but the mechanism of changing volume due to the loss of water present in the mixes over time. Therefore, there is the possibility of observing higher drying shrinkage value for the mixes, in which more water is available inside the mixes to get evaporated over time. Drying shrinkage was determined based on the change of length in longer dimensions of the cast specimens. In the present work, cast specimens cured at ambient temperature and at 80°C for 24 hours followed by ambient temperature were studied for the determination of drying shrinkage of the produced FA based geopolymer mortar mixes, as these mixes cured under these two curing regimes had shown better strength. In the following sections, effect of RFA, concentration of SH solution in AL, SS/SH ratio in AL and the AL/FA ratio on the drying shrinkage of the cast specimens cured at different curing regimes has been discussed.



SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.30 Variation of drying shrinkage value for different mixes produced with AL having 6M SH solution and AL/FA = 0.4

4.5.1 Ambient Temperature Curing

No significant variation was found in the change of length for FA based geopolymer mortar mixes after 28 days of ambient temperature curing. It has been observed that maximum length change was observed at 28 days of curing. Figure 4.30 - 4.41 represents the variation profiles of the observed drying shrinkage value of the cast samples of various FA based geopolymer mortar mixes cured at ambient temperature.

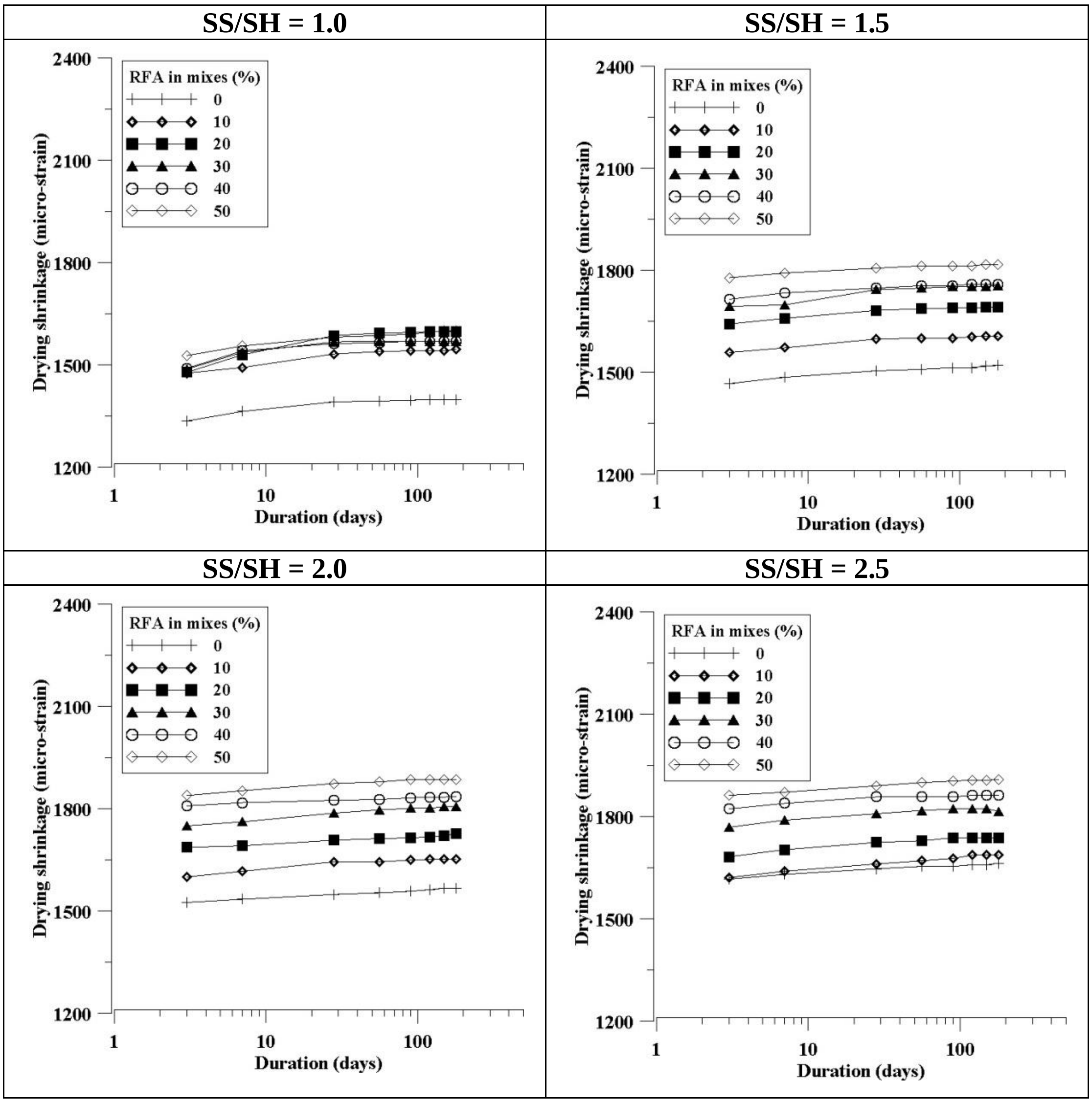


Figure 4.31 Variation of drying shrinkage value for different mixes produced with AL having 8M SH solution and AL/FA = 0.4

4.5.1.1 Effect of RFA content

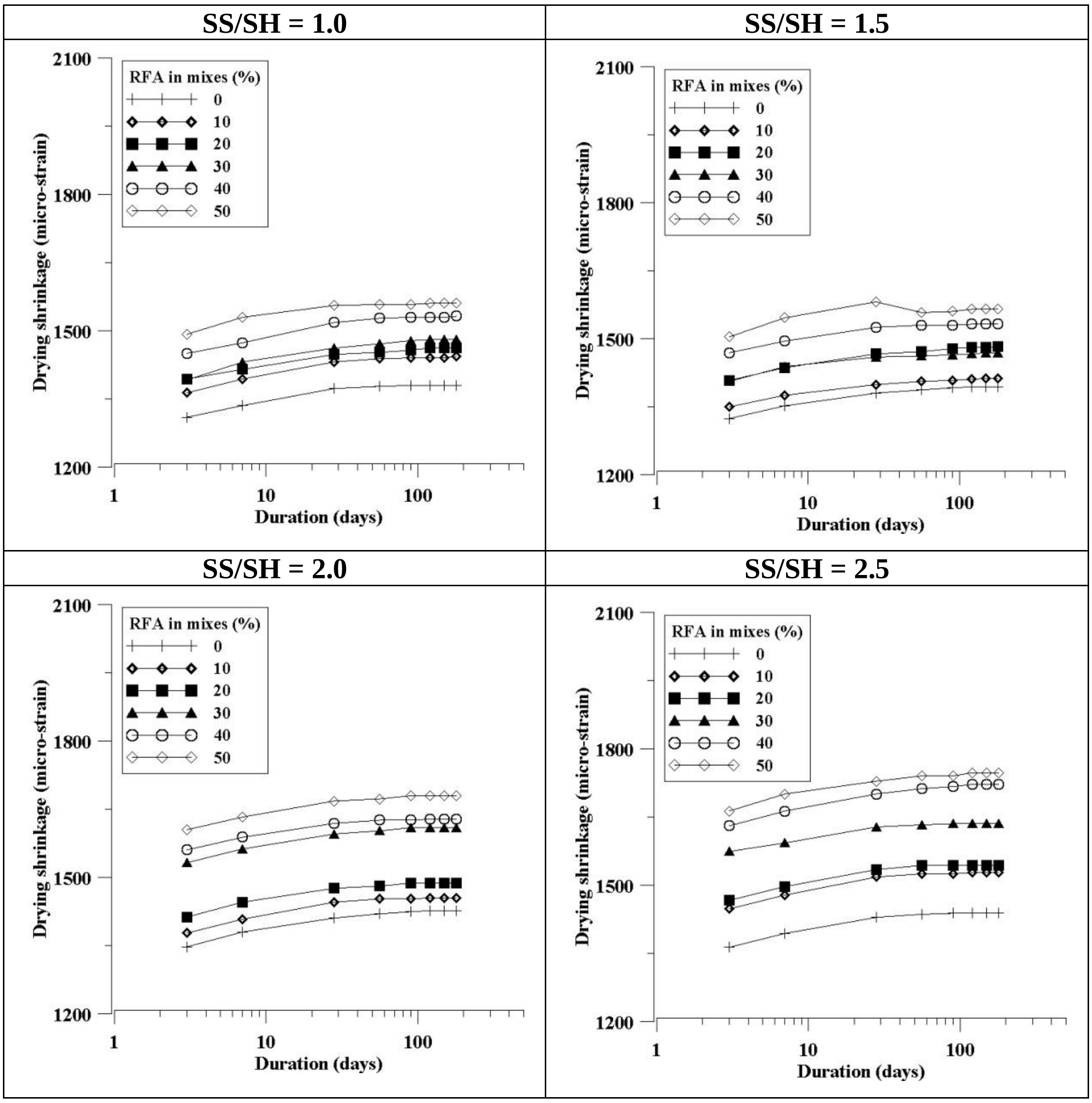


Figure 4.32 Variation of drying shrinkage value for different mixes produced with AL having 10M SH solution and AL/FA = 0.4

More change in length of the cast specimens i.e. higher drying shrinkage value was observed for the mortar mixes having higher RFA content for ambient temperature curing. Higher water absorption capacity, the lesser density of RFA etc. is the key reasons to exhibit higher change in length of the cast specimens. Higher content of RFA absorbs more water and over time, this water can be evaporated from the samples. Higher drying shrinkage value or change in longer dimension of the cast specimenswas observed for the mortar mixes produced with 50% RFA than that of mortar mixes produced with no RFA. The average higher drying shrinkage value was found to be 18% and 8.1% for the mixes having 50% of RFA than that of mixes with no RFA produced with AL/FA ratio of 0.4 and 0.6 respectively for all the combinations when samples were cured at ambient temperature.

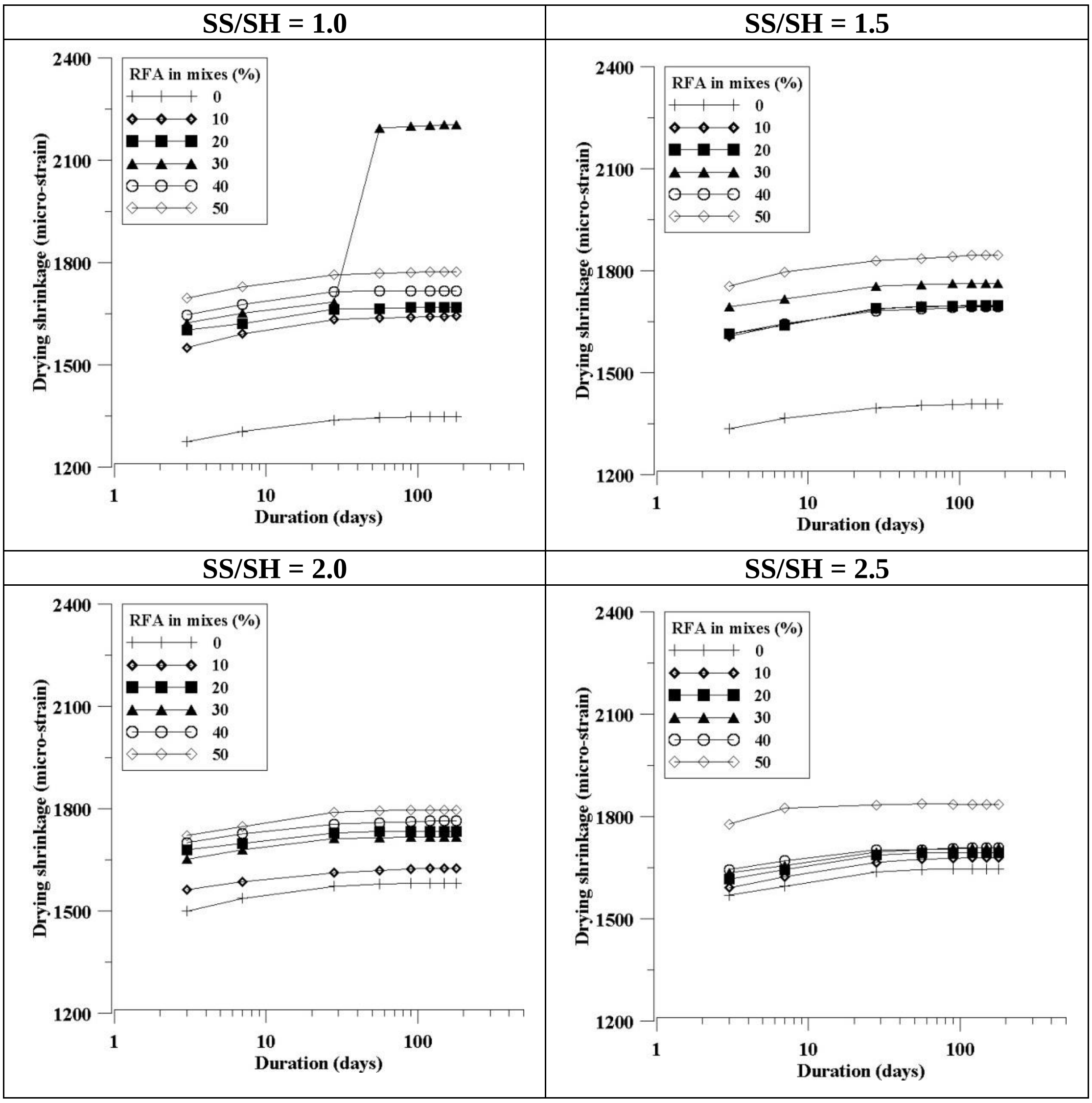


Figure 4.33 Variation of drying shrinkage value for different mixes produced with AL having 12M SH solution and AL/FA = 0.4

4.5.1.2 *Effect of Concentration of SH solution*

Significantchange in length in longer dimension was observed for the cast samples of different FA based geopolymer mortar mixes which were produced with AL having lower concentration of SH solution in it. As the concentration of SH solution in AL increases, the quantity of water in total AL decreases. Therefore, mortar mixes produced with AL having higher concentration SH solution may not have more water molecules, which can be evaporated over time. As results, lower drying shrinkage value was observed for the mortar mixes produced with higher concentration of SH solution. Approximately average 20.9% and 22.2% lower drying shrinkage value was observed for the mixes produced with AL having 16M SH solution than that of mixes produced with AL having 6M SH solution for AL/FA=0.4 and 0.6 respectively for ambient temperature curing.

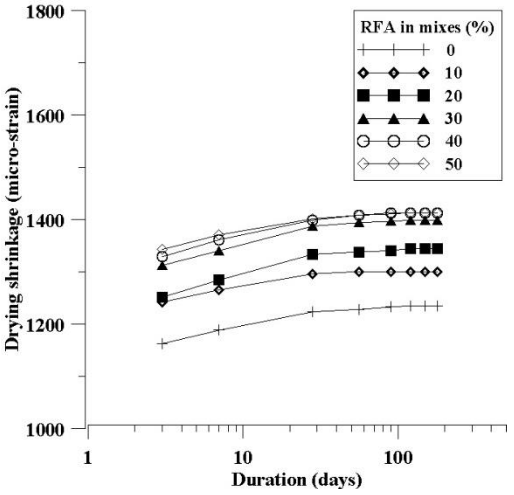
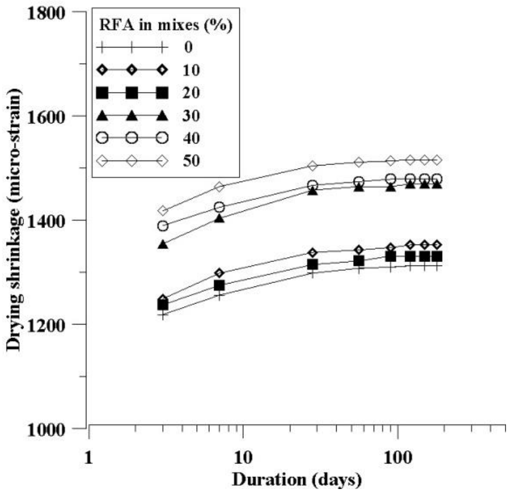
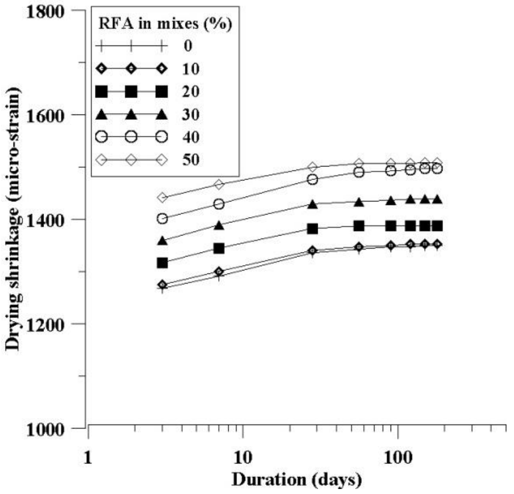
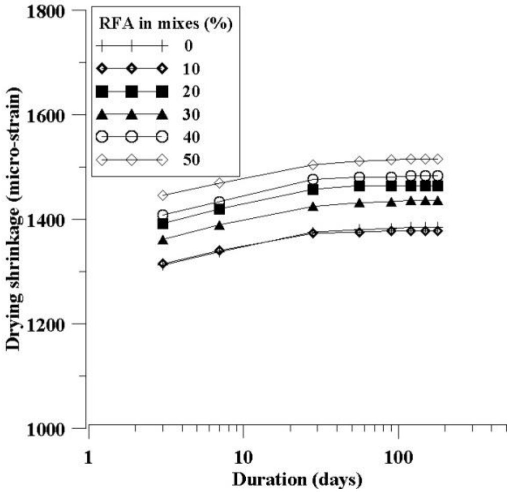
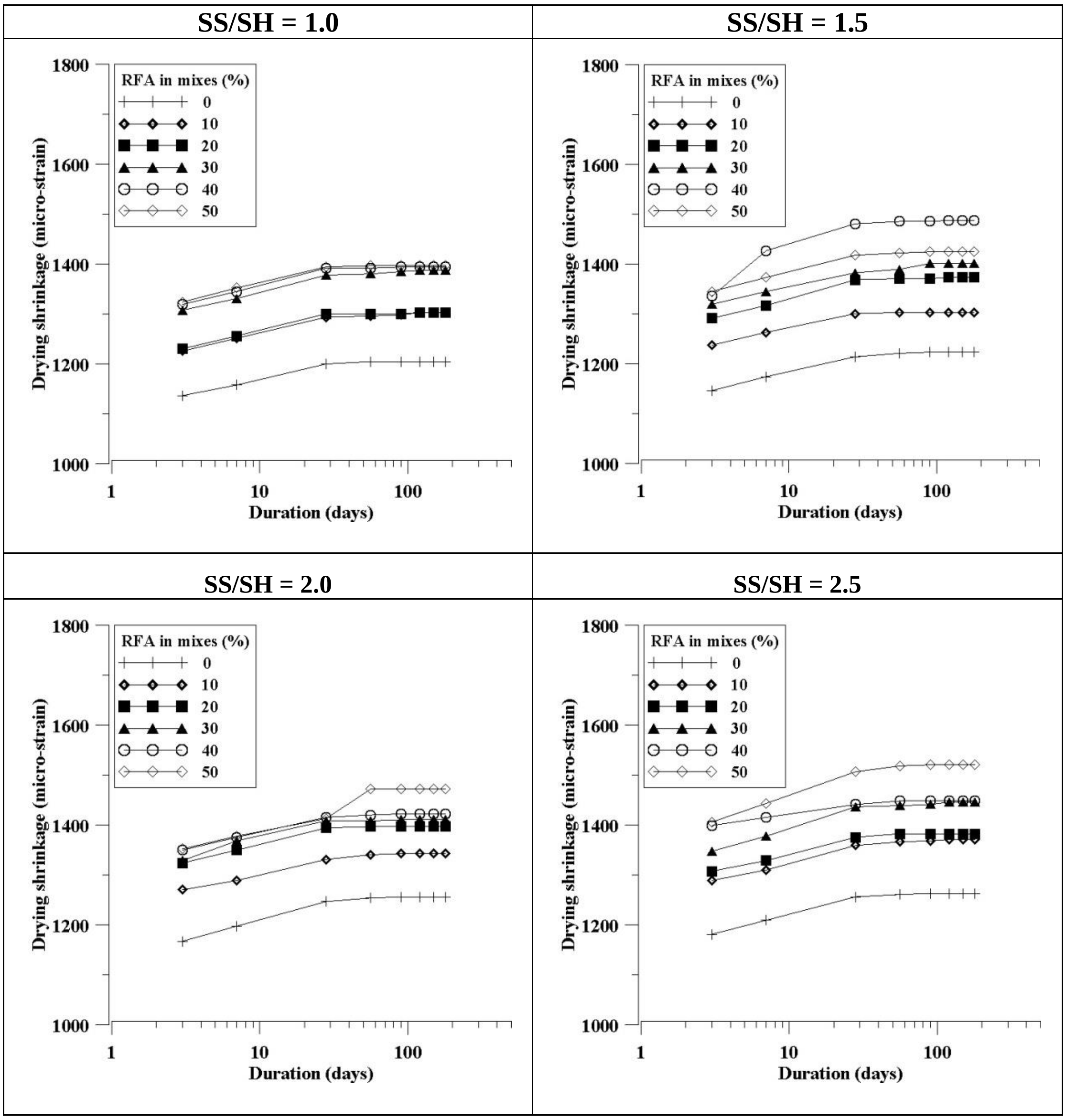
SS/SH = 1.0	**SS/SH = 1.5**
	
SS/SH = 2.0	**SS/SH = 2.5**
	

Figure 4.34 Variation of drying shrinkage value for different mixes produced with AL having 14M SH solution and AL/FA = 0.4


SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.35 Variation of drying shrinkage value for different mixes produced with AL having 16M SH solution and AL/FA=0.4

4.5.1.3 Effect of SS/SH ratio

Higher drying shrinkage value was found for the mortar mixes produced with AL consisting of less SH solution. If SS/SH ratio increases in total AL for a particular concentration of SH solution, the quantity of water in total AL gets increased because of SS solution used consists of approximately 63.5% water by mass. Cast mortar samples cured at ambient temperature and produced with the ratio of SS solution to SH solution 2.5 exhibited average 11.6% and 7.3% higher shrinkage value than that of the cast mortar samples produced with the ratio of SS solution to SH solution 1.0 while producing mortar mixes with AL/FA = 0.4 and 0.6 respectively.

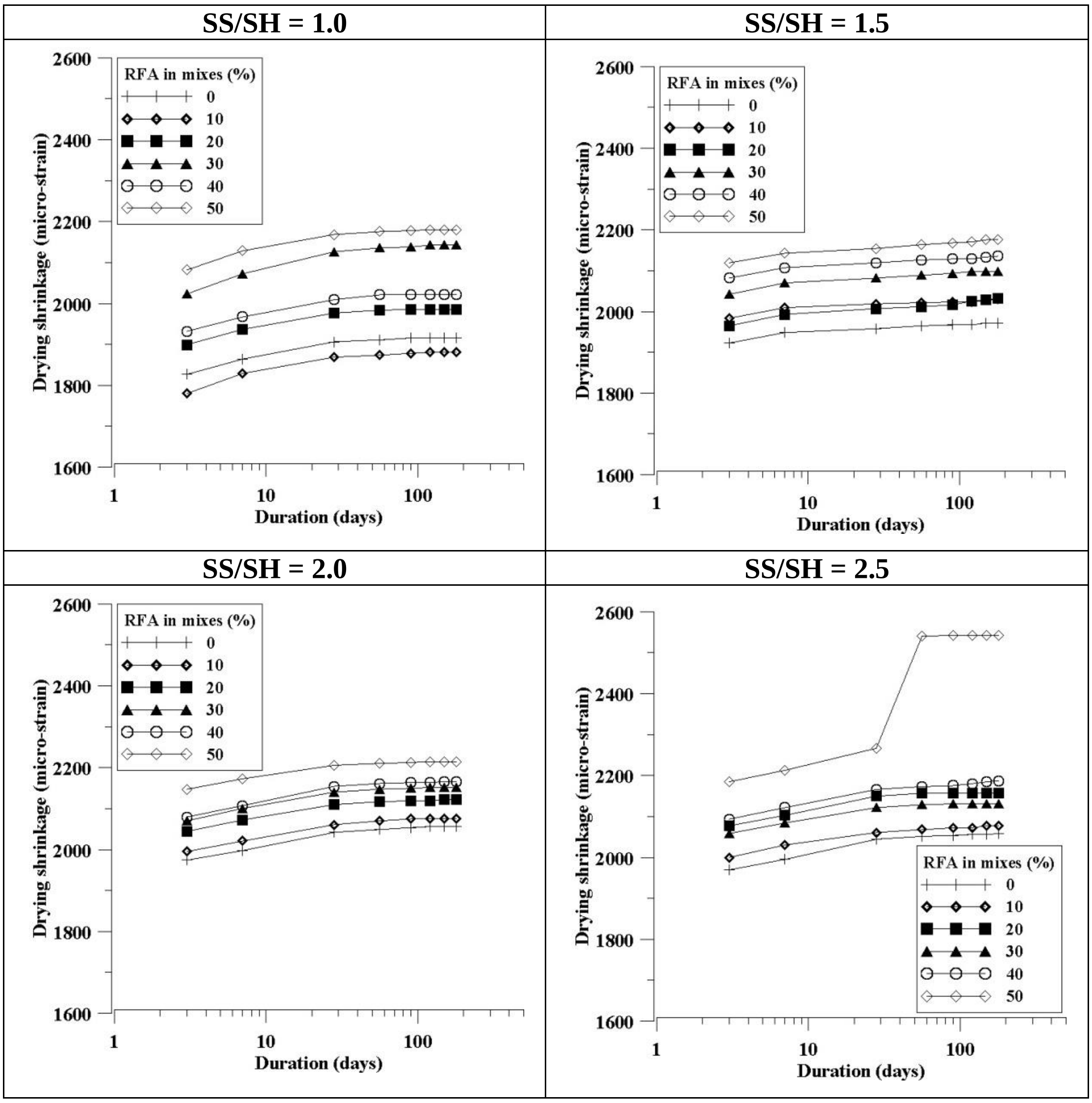


Figure 4.36 Variation of drying shrinkage value for different mixes produced with AL having 6M SH solution and AL/FA=0.6

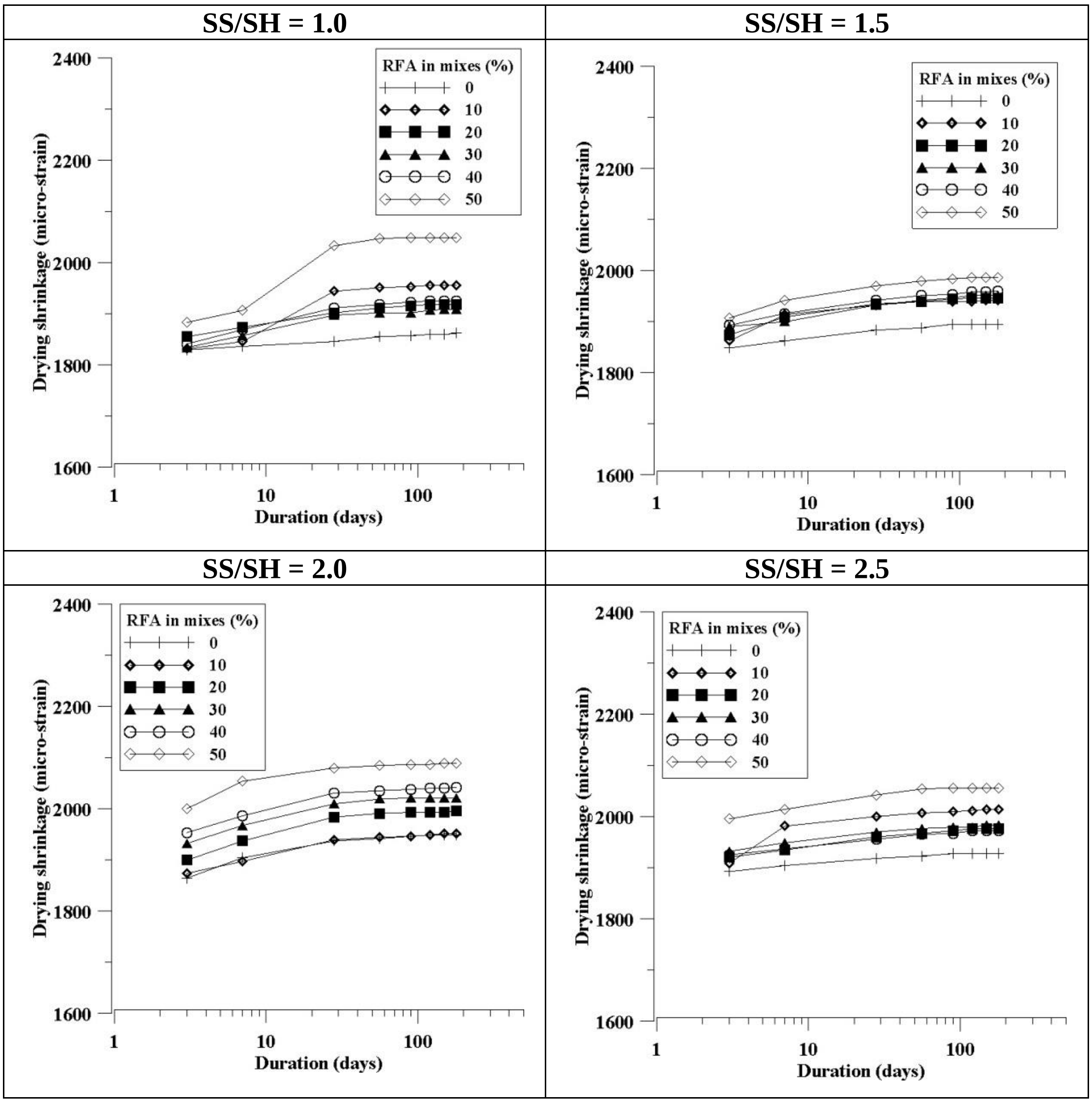


Figure 4.37 Variation of drying shrinkage value for different mixes produced with AL having 8M SH solution and AL/FA=0.6

4.5.1.4 *Effect of AL/FA ratio*

From the experimental observations, lower drying shrinkage values were observed for the mortar mixes produced with lower AL/FA ratio. Mortar mixes produced with AL/FA = 0.6 was found to be showing higher drying shrinkage value than mortar mixes produced with AL/FA = 0.4. Water quantity in mortar mixes is increased, when AL/FA ratio was adopted higher. Therefore, there wasa high chance to evaporate more quantity of water from the cast samples and higher change in length in the direction of longer dimension and volume as well which leads to higher contraction of the cast samples for the mortar mixes produced with AL/FA ratio of 0.6. Even though with the increment of AL/FA ratio, drying shrinkage value of the cast samples increased, the average increment of drying shrinkage value with respect to other parameters was being observed relatively lesser when higher AL/FA was adopted to produce the mortar mixes.

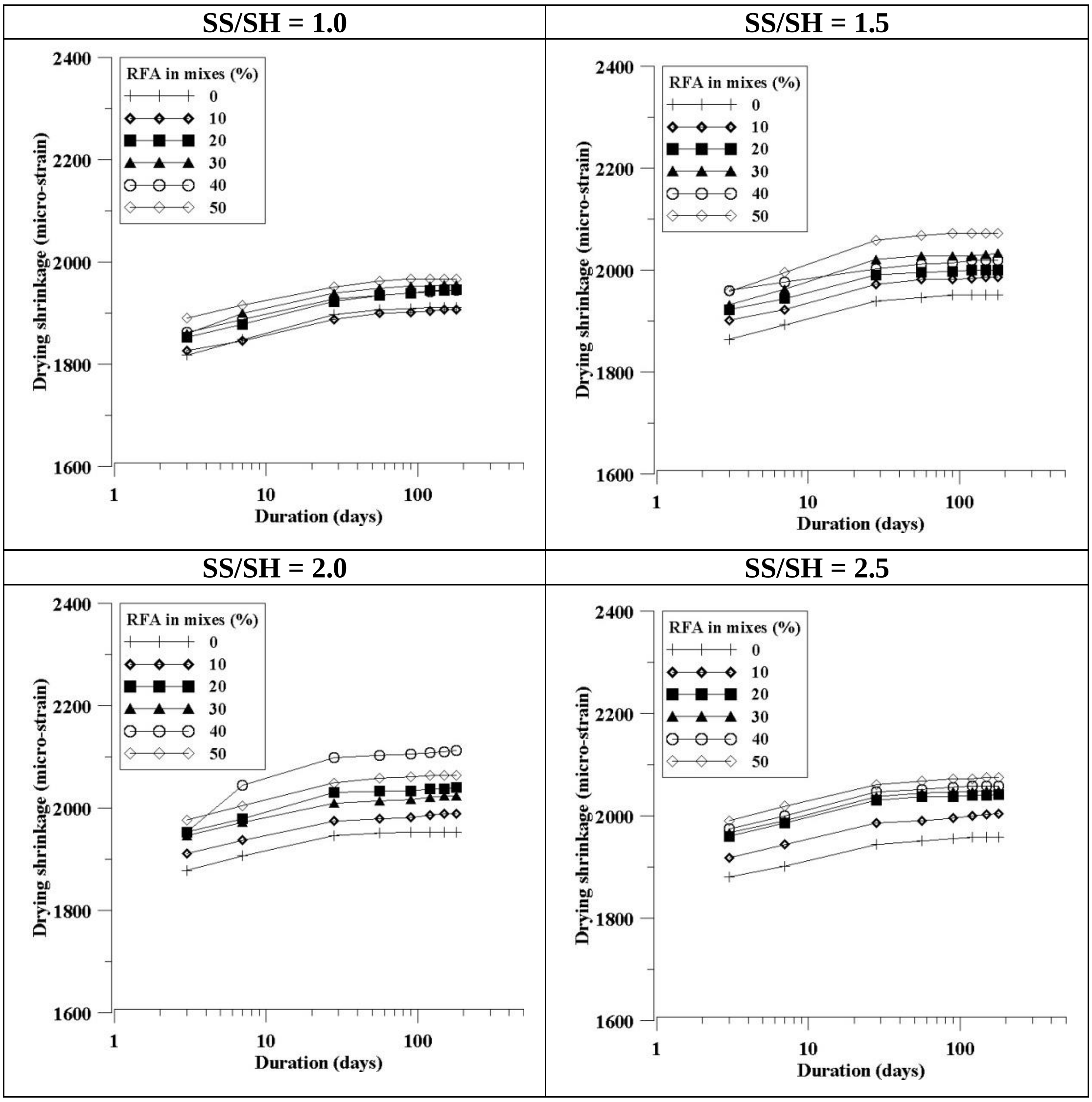


Figure 4.38 Variation of drying shrinkage value for different mixes produced with AL having 10M SH solution and AL/FA=0.6

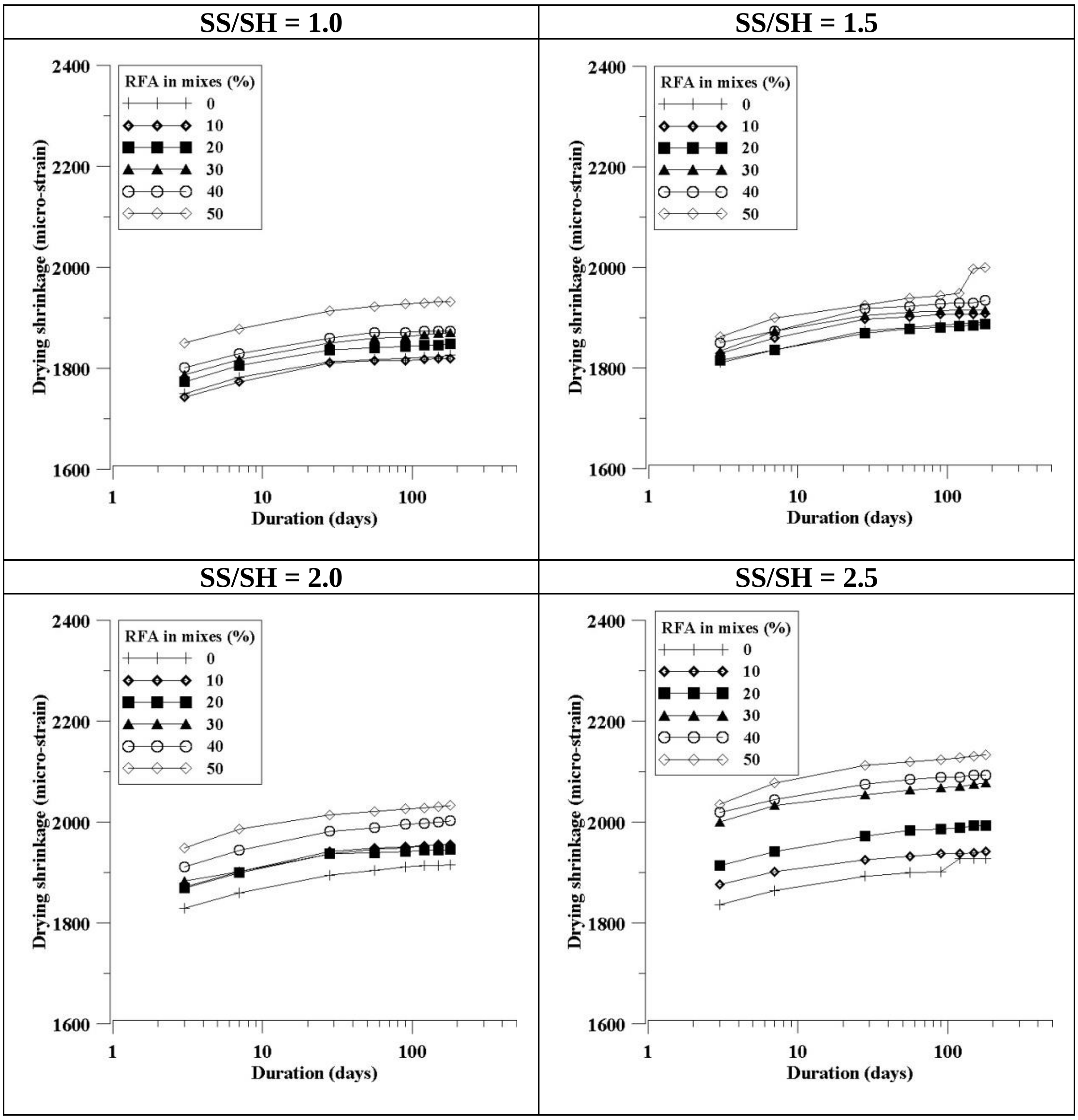


Figure 4.39 Variation of drying shrinkage value for different mixes produced with AL having 12M SH solution and AL/FA=0.6

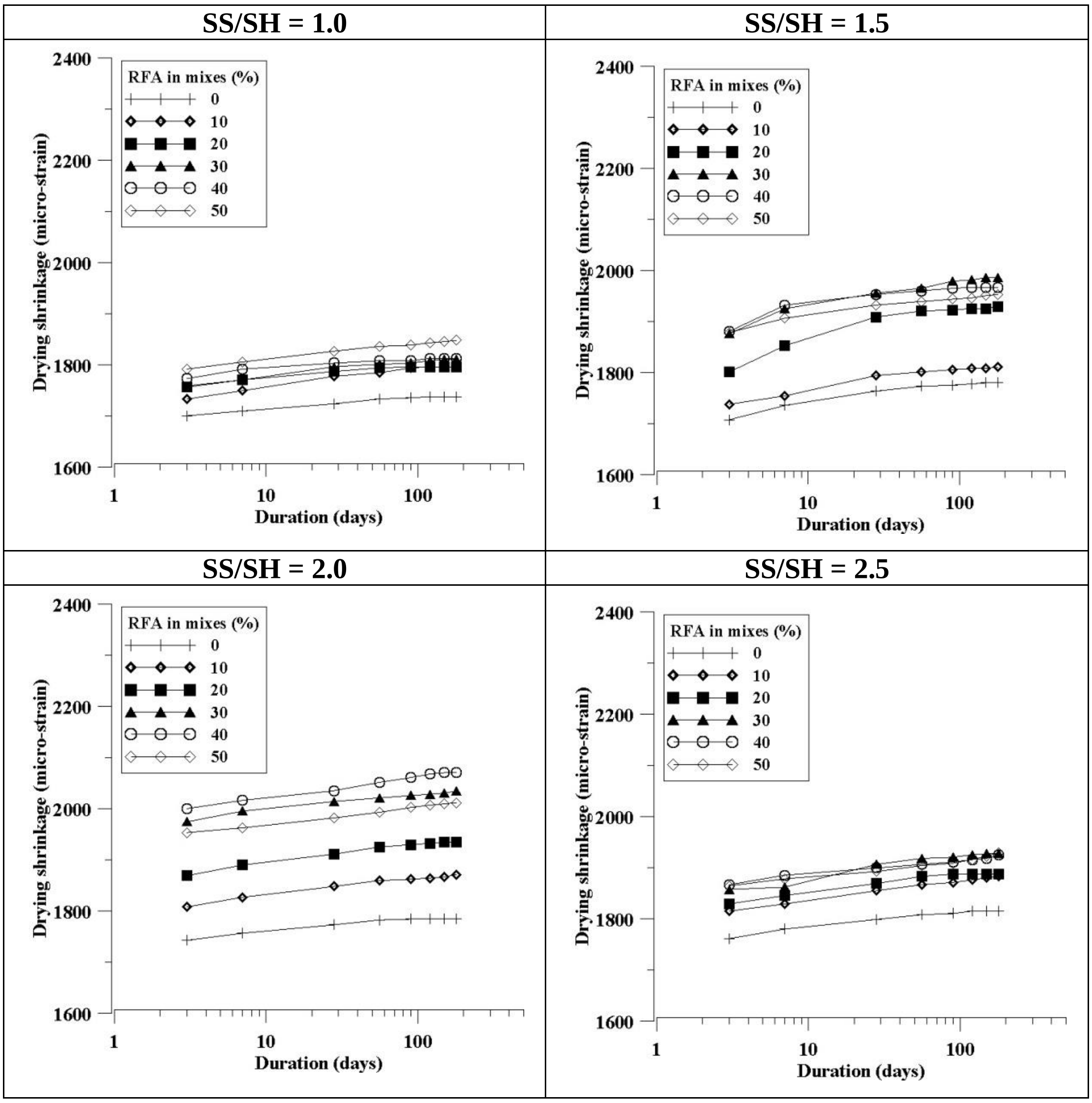


Figure 4.40 Variation of drying shrinkage value for different mixes produced with AL having 14M SH solution and AL/FA=0.6

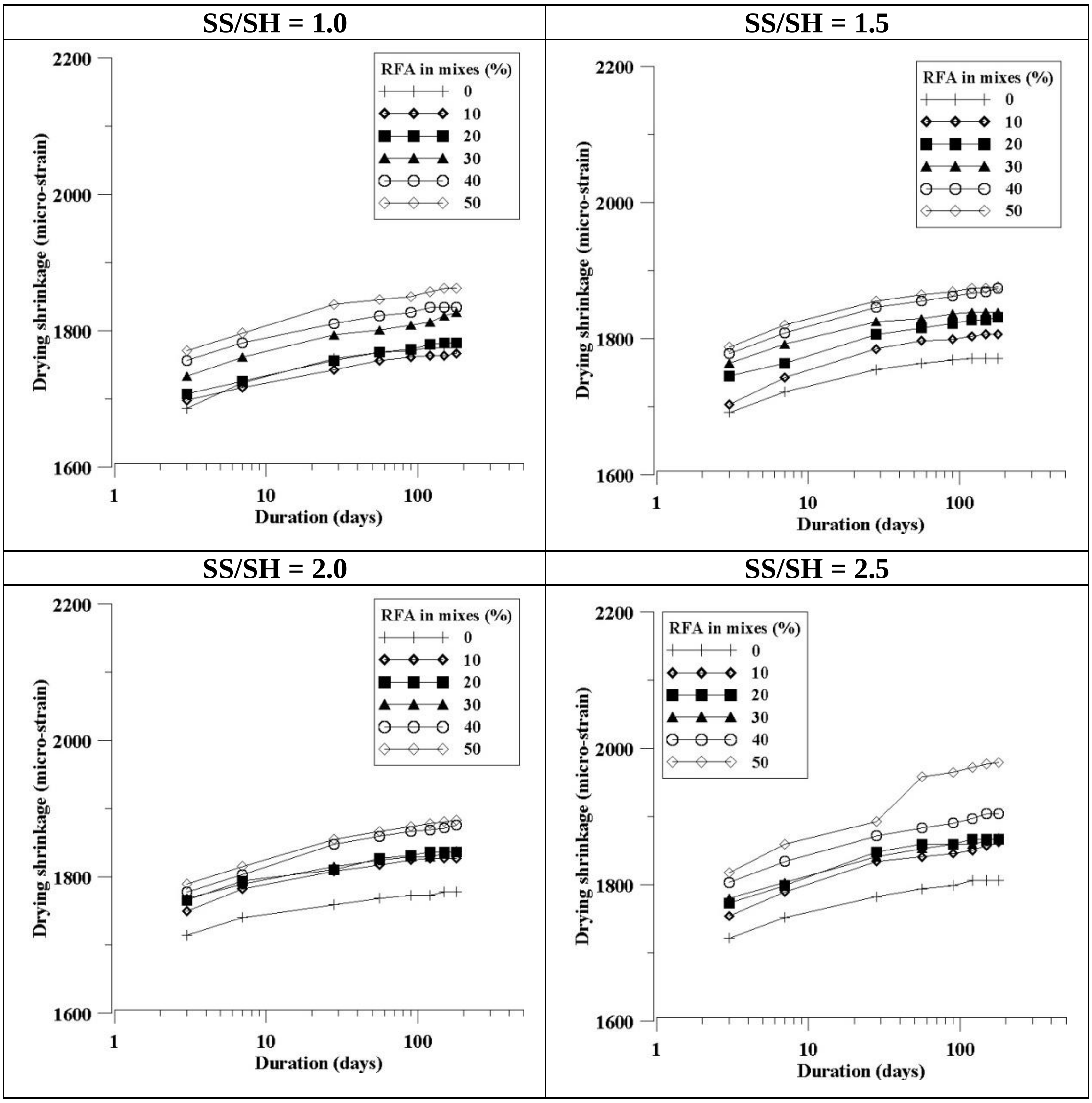


Figure 4.41 Variation of drying shrinkage value for different mixes produced with AL having 16M SH solution and AL/FA=0.6

4.5.2 Heat Curing

In the present study, there was no such significant variation found in the drying shrinkage for FA based geopolymer mortar mixes after longer duration when samples were heat cured for specified duration. It was observed that maximum drying shrinkage occurred in the early duration only at the age of 3 days. During heat curing, most of the AL reacts with FA to produce geopolymeric product at a faster rate and as well some quantity water also evaporated. Water molecules inside the samples may not be present to get evaporated and as a result, significant shrinkage had not been observed after 3 days. Figure 4.42 - 4.53 represents the variation profiles of the observed drying shrinkage value of the samples of various FA based geopolymer mortar mixes cured at 80°C for 24 hours.

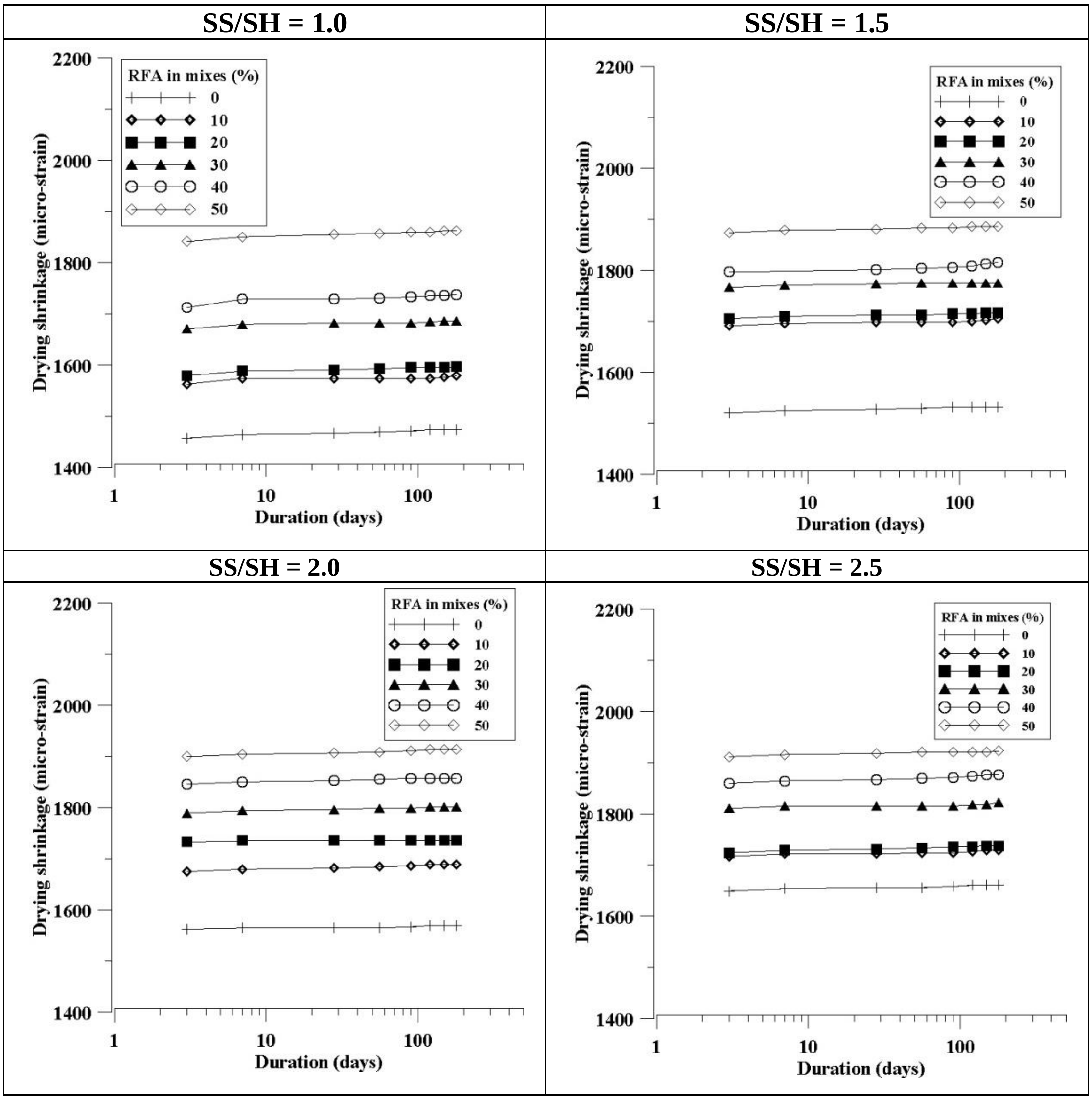
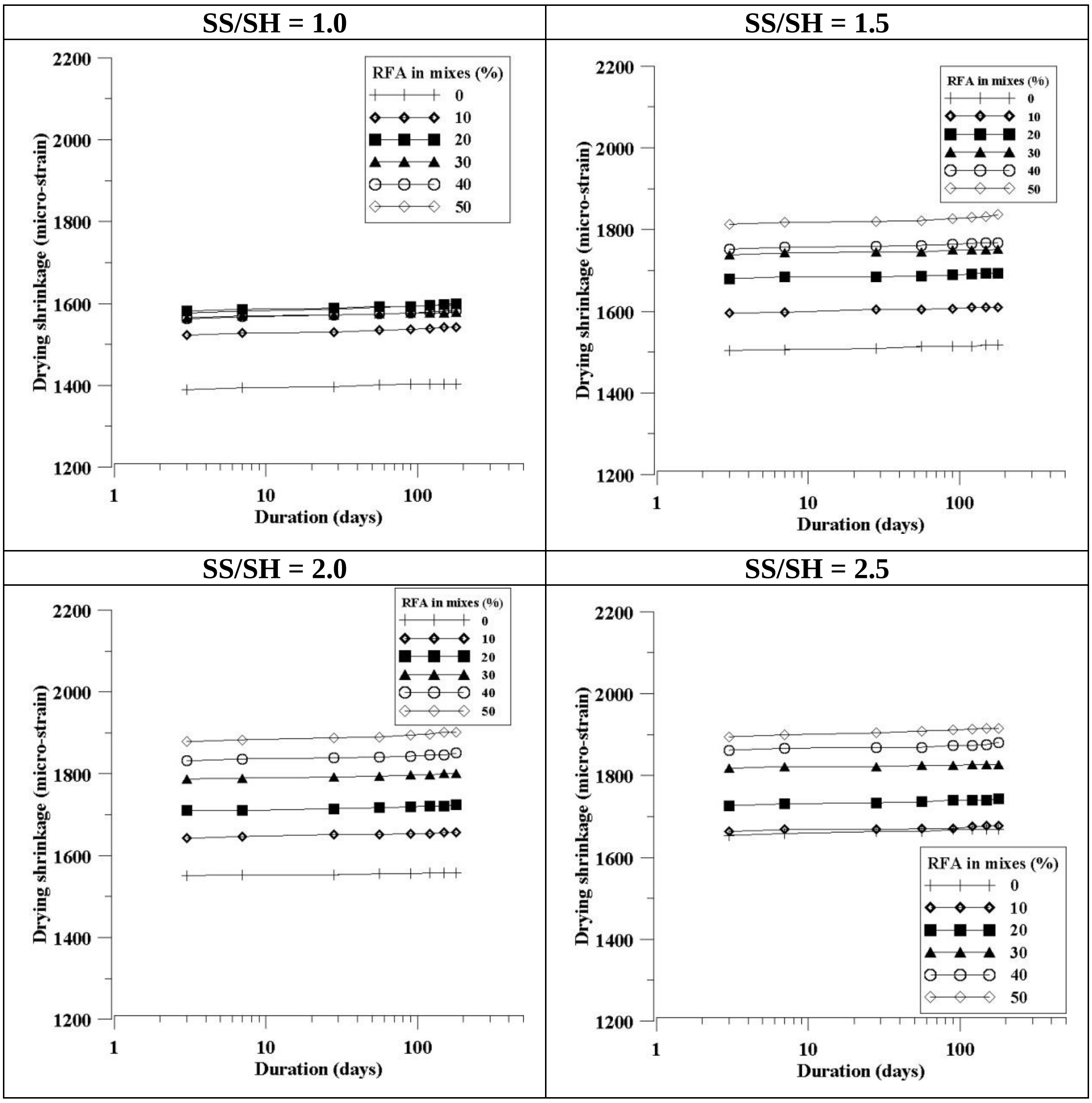


Figure 4.42 Variation of drying shrinkage value for different mixes produced with AL having 6M SH solution and AL/FA=0.4


SS/SH = 1.0

SS/SH = 1.5

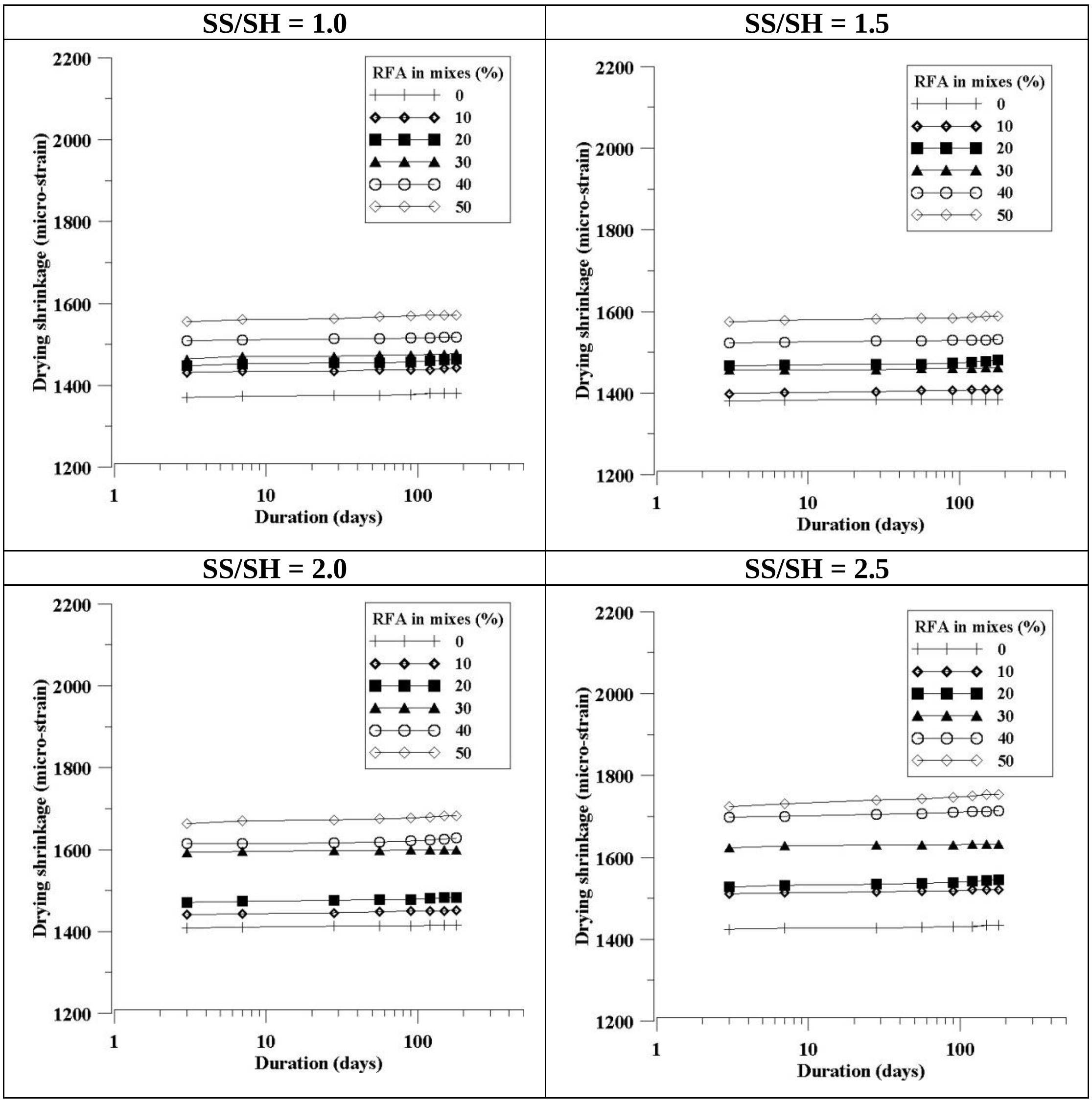
SS/SH = 2.0

SS/SH = 2.5

Figure 4.43 Variation of drying shrinkage value for different mixes produced with AL having 8M SH solution and AL/FA=0.4

4.5.2.1 Effect of RFA content

Less change in length of the cast specimens i.e. lesser drying shrinkage value was observed for the mortar mixes having lesser RFA content for heat curing. This observation may be due to the higher water absorption capacity, lesser density of RFA etc. Higher content of RFA absorbs more water and over time, this water can be evaporated from the samples. Higher drying shrinkage value or change in longer dimensionwas observed for the mortar mixes produced with 50% RFA than that of mortar mixes produced with no RFA. When prism samples were cured at 80°C for 24 hours, average 19% and 7% higher drying shrinkages value were found for the mixes having 50% RFA than that of mixes with no RFA for all the combinations for AL/FA=0.4 and 0.6 respectively.



SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.44 Variation of drying shrinkage value for different mixes produced with AL having 10M SH solution and AL/FA = 0.4

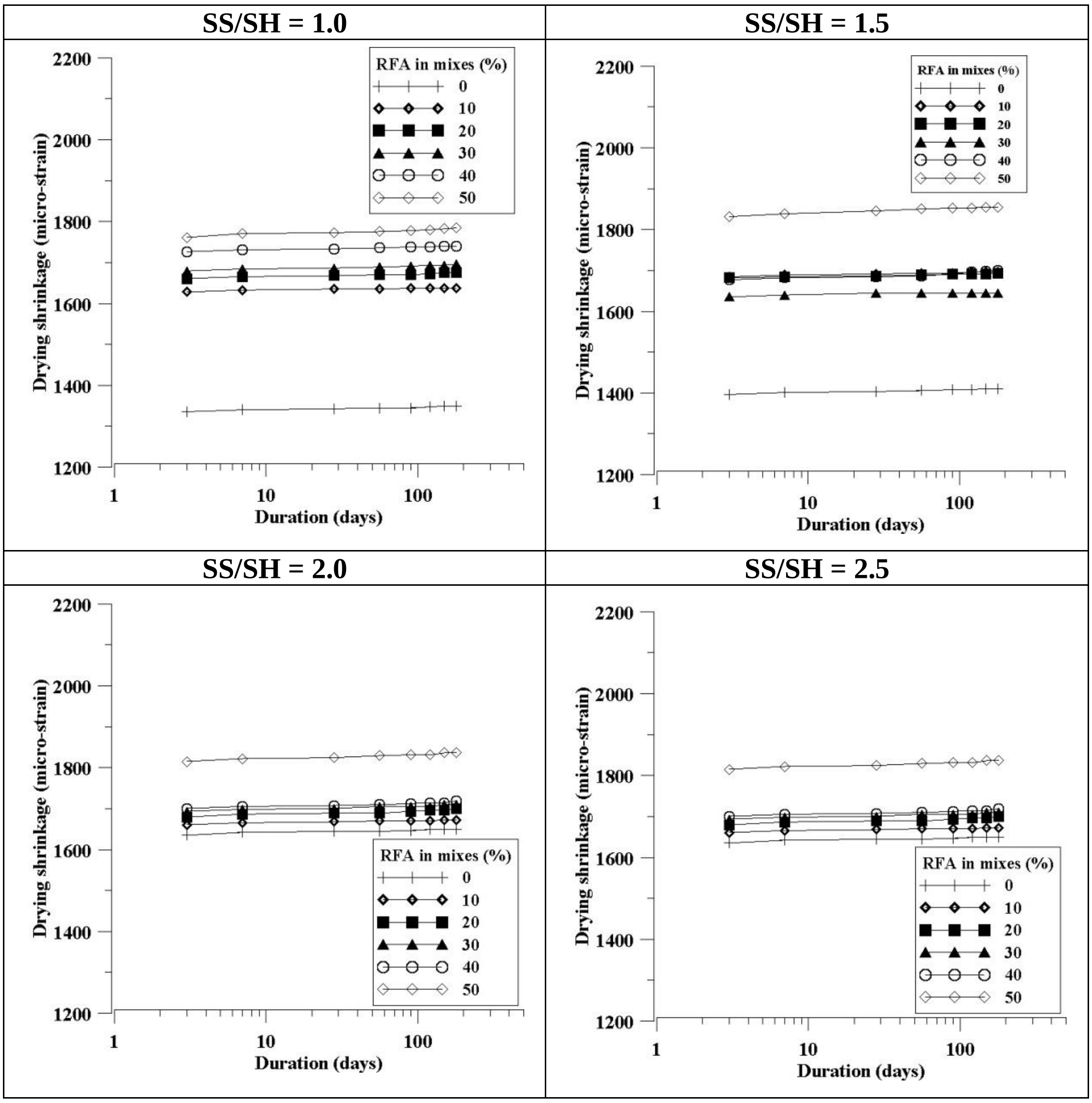


Figure 4.45 Variation of drying shrinkage value for different mixes produced with AL having 12M SH solution and AL/FA = 0.4

4.5.2.2 Effect of Concentration of SH solution

Higher drying shrinkage value was observed for the mortar mixes, which were produced with AL having lower concentration of SH solution in it. As the concentration of SH solution in AL increases, the quantity of water in total AL decreases. Therefore, mortar mixes produced with higher concentration SH solution does not have more water molecules, which can be evaporated. As results, lower drying shrinkage was observed for the mortar mixes produced with higher concentration of SH solution. Water molecules inside the samples may not be present to get evaporated and as a result, significant shrinkage had not been observed after 3 days. For AL/FA = 0.4 and 0.6, average 19% and 17% lower drying shrinkage value was observed for the mixes produced with AL having 16M SH solution than that of the mortar mixes produced with AL having 6M SH solution respectively.

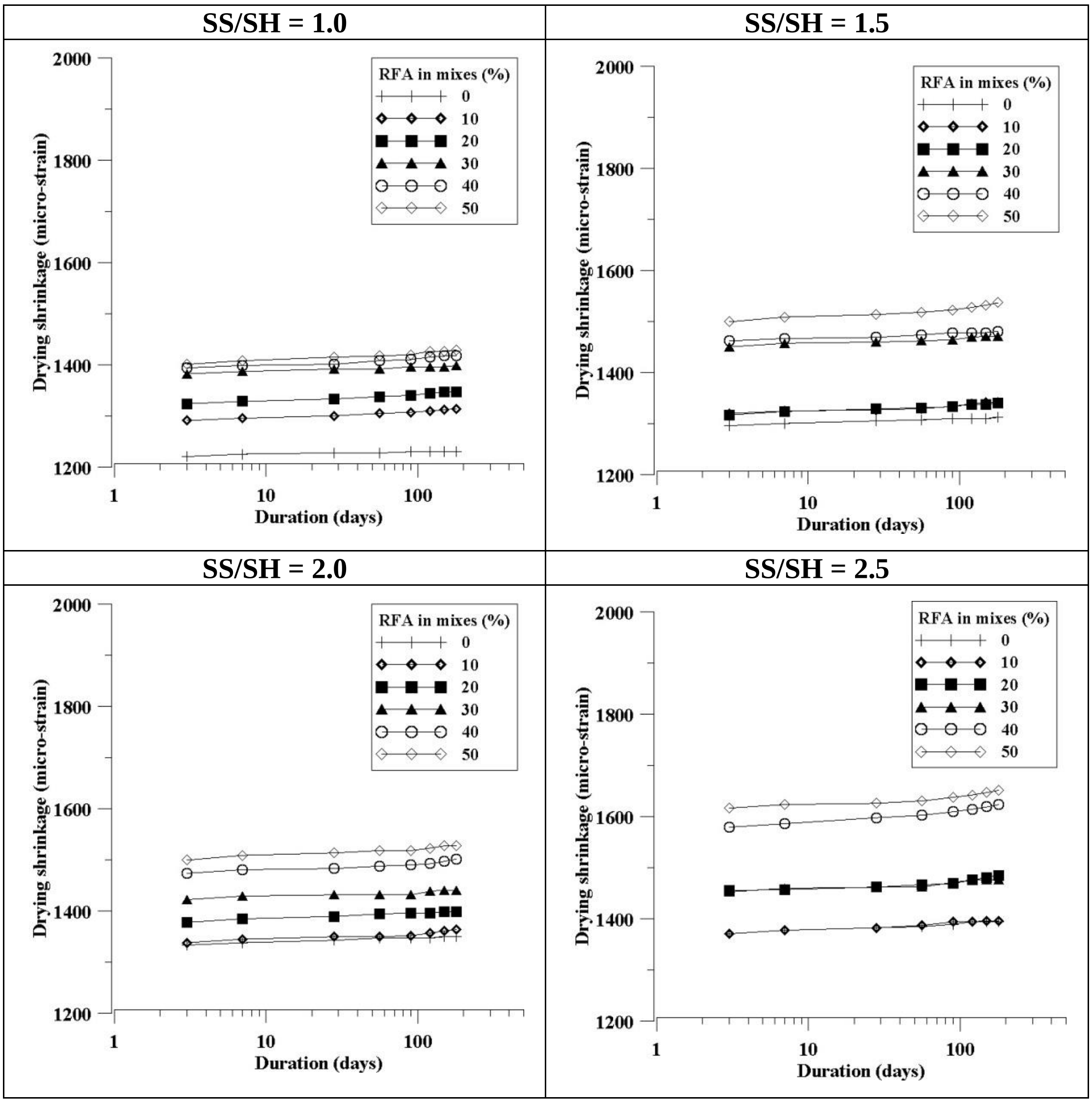
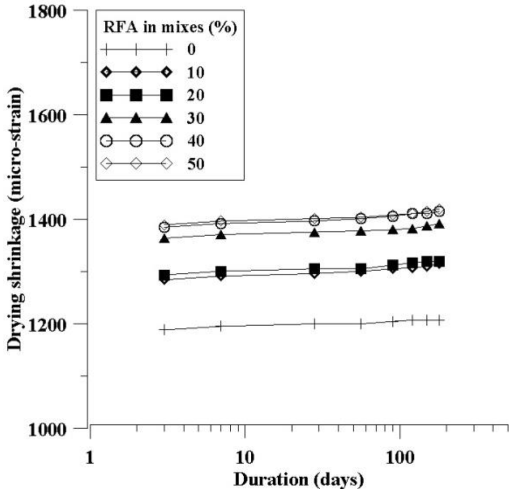
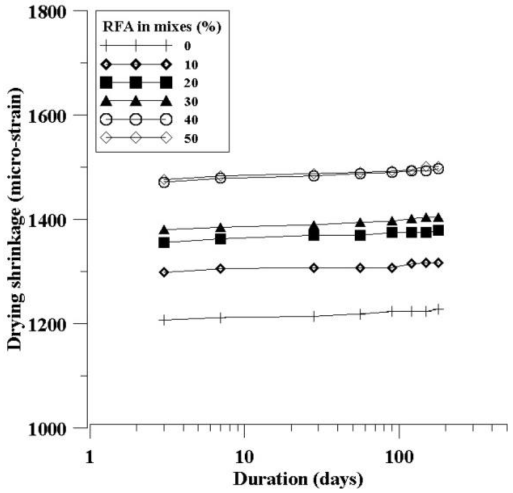


Figure 4.46 Variation of drying shrinkage value for different mixes produced with AL having 14M SH solution and AL/FA = 0.4

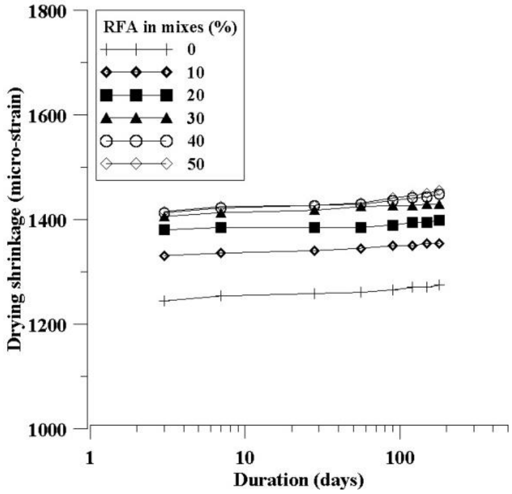
SS/SH = 1.0



SS/SH = 1.5



SS/SH = 2.0



SS/SH = 2.5

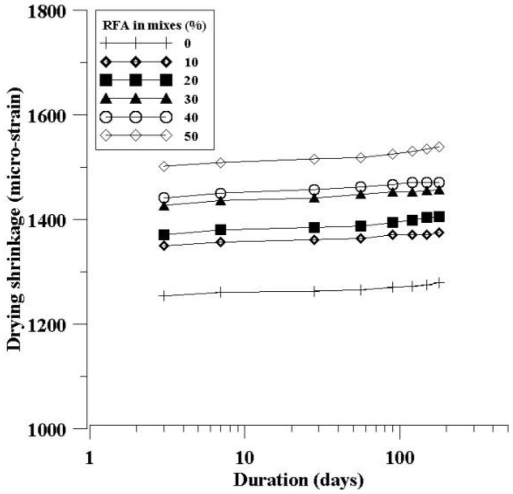
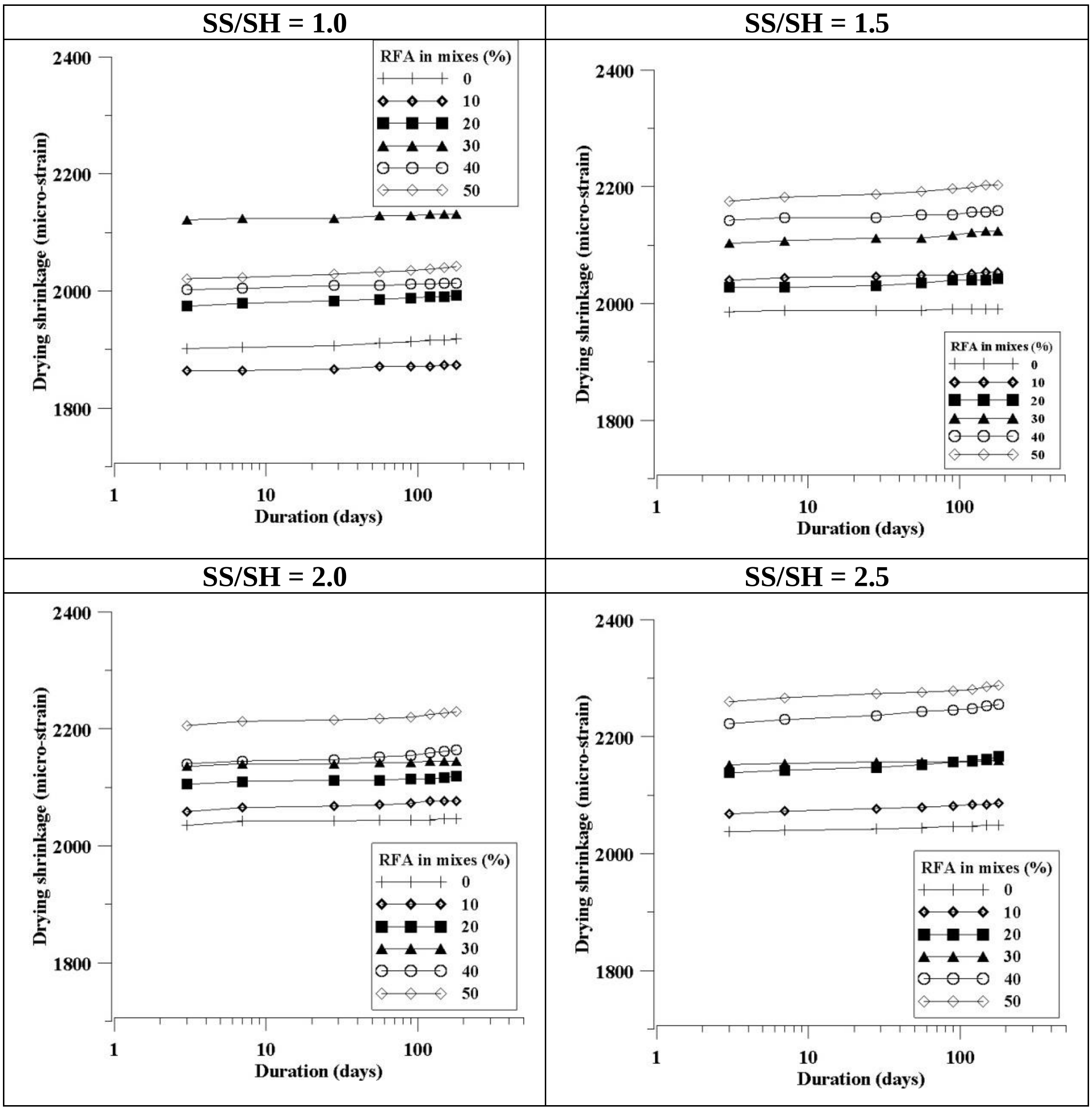


Figure 4.47 Variation of drying shrinkage value for different mixes produced with AL having 16M SH solution and AL/FA=0.4


SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.48 Variation of drying shrinkage value for different mixes produced with AL having 6M SH solution and AL/FA=0.6

4.5.2.3 Effect of SS/SH ratio in AL

Higher drying shrinkage value was observed for the mortar mixes produced with AL consisting of less SH solution for heat cured samples. If the ratio of SS solution to SH solution increases in total AL for a particular concentration of SH solution, the quantity of water in total AL gets increased because of SS solution used consists of approximately 63.5% water by mass. An average higher shrinkagevalueof7.2% and 6% for cast mortar samples cured at 80°C for 24 hours was found for the mortar mixes produced with the SS/SH ratio of 2.5 than that of the mortar mixes produced with the SS/SH ratio of 1.0 for AL/FA= 0.4 and 0.6 respectively.

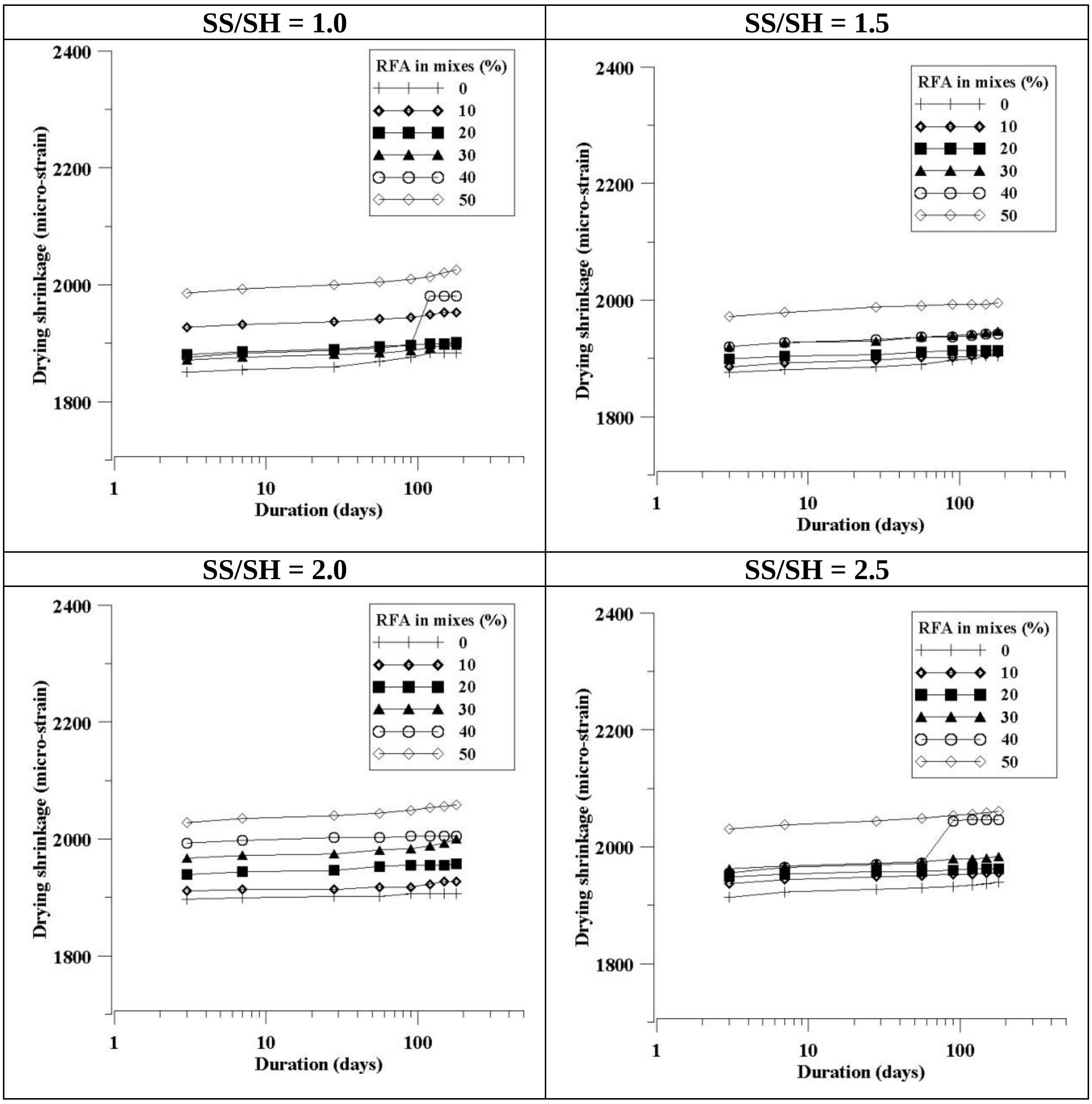
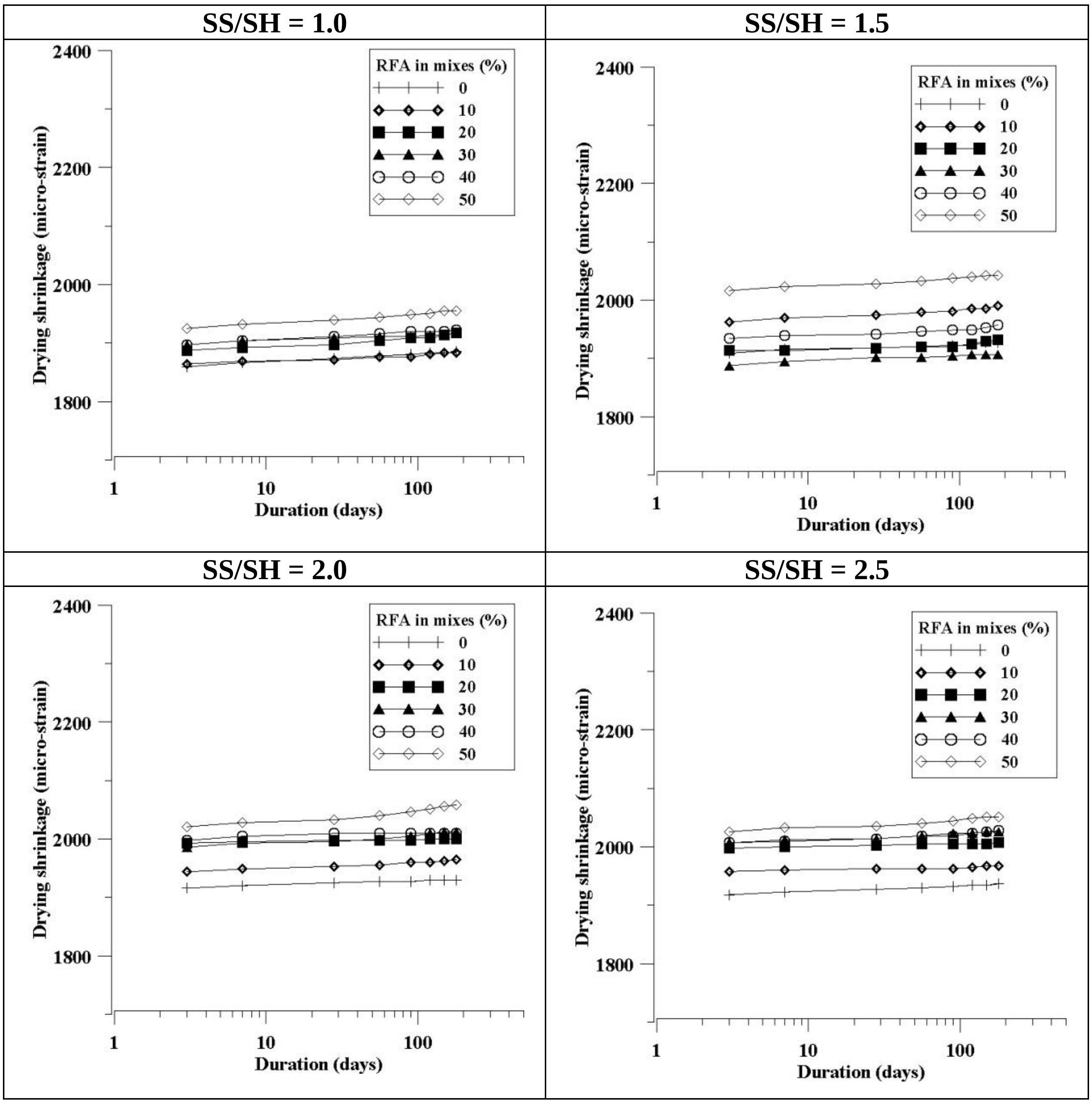


Figure 4.49 Variation of drying shrinkage value for different mixes produced with AL having 8M SH solution and AL/FA=0.6

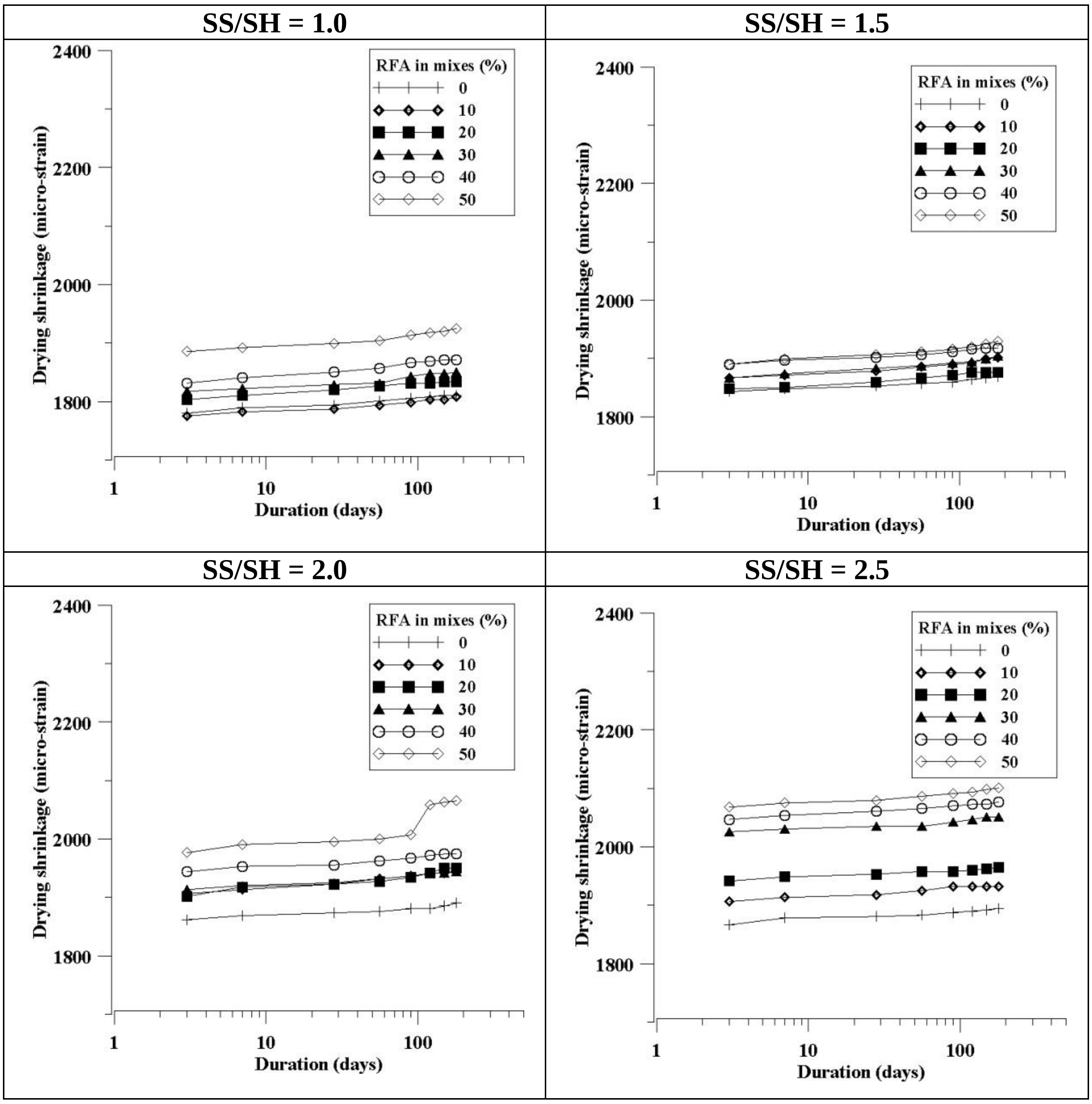

SS/SH = 1.0

SS/SH = 1.5

SS/SH = 2.0

SS/SH = 2.5

Figure 4.50 Variation of drying shrinkage value for different mixes produced with AL having 10M SH solution and AL/FA=0.6


SS/SH = 1.0

SS/SH = 1.5

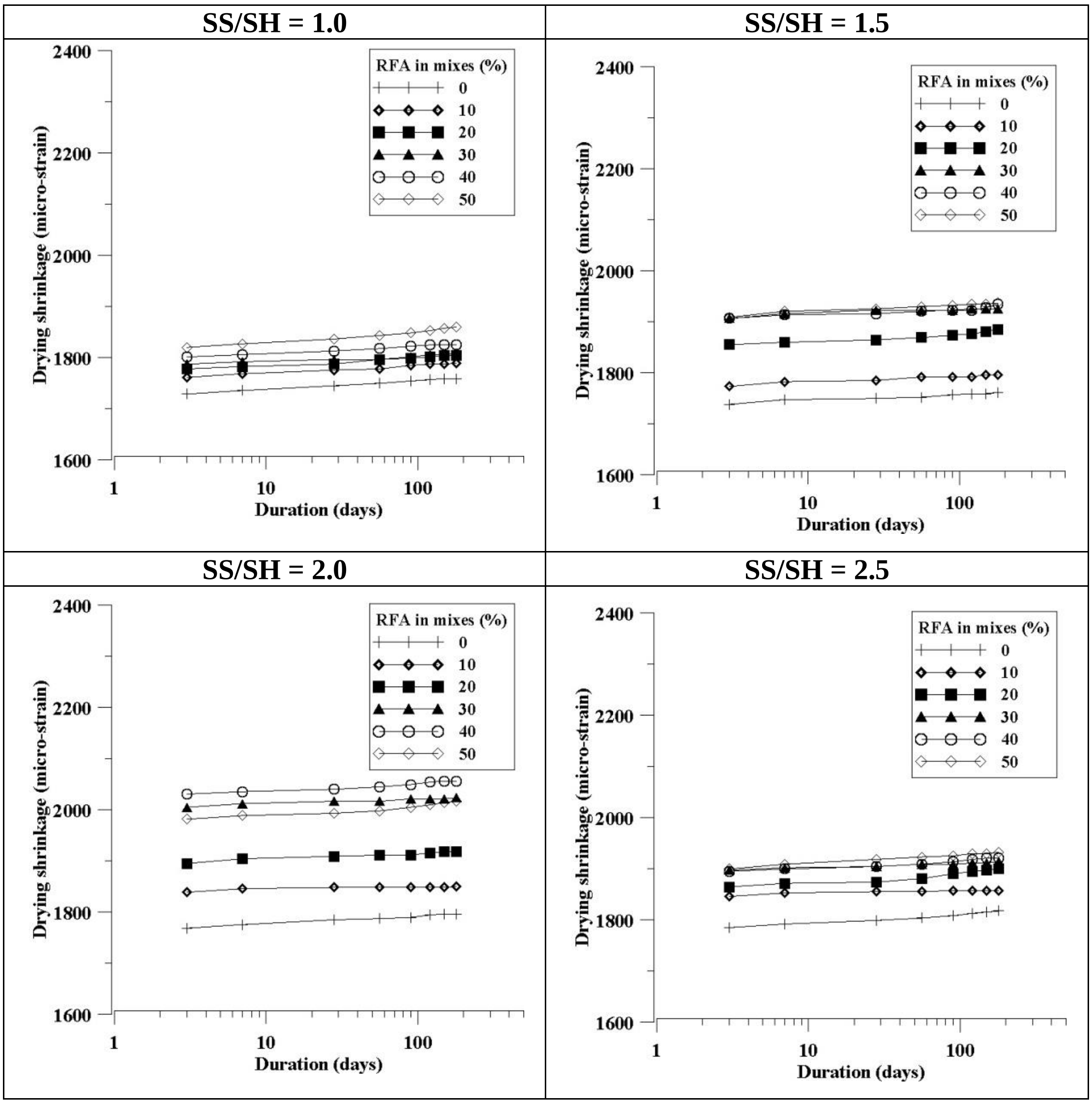
SS/SH = 2.0

SS/SH = 2.5

Figure 4.51 Variation of drying shrinkage value for different mixes produced with AL having 12M SH solution and AL/FA=0.6

4.5.2.4 Effect of AL/FA ratio

From the experimental observations, lower drying shrinkage values are observed for the mortar mixes produced with lower AL/FA ratio. Mortar mixes produced with AL/FA = 0.6 were found to be showing higher drying shrinkage value than mortar mixes produced with AL/FA = 0.4. Water Quantity in mortar mixes was increased when AL/FA ratio was adopted higher. Therefore, there washigh chance to evaporate more quantity of water from the cast samples and higher change in length in the direction of longer dimension and volume as well, which leads to higher contraction of the cast samples for the mortar mixes produced with AL/FA ratio of 0.6. Even though with the increment of AL/FA ratio, drying shrinkage value of the cast samples increased, relatively average increment of drying shrinkage value with respect to other parameters was being observed lesser when higher AL/FA was adopted to produce the mortar mixes. It may be found because AL reacts with FA to form geopolymeric matrix, such that free water molecules will not get evaporated easily from the cast samples.


SS/SH = 1.0

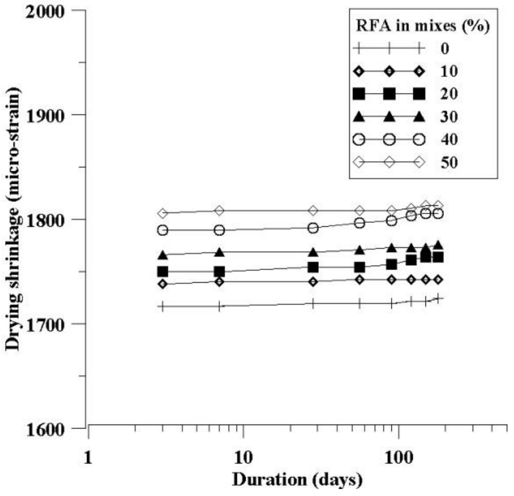
SS/SH = 1.5

SS/SH = 2.0

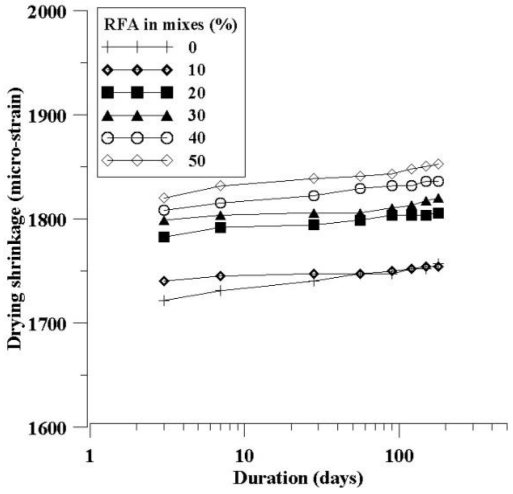
SS/SH = 2.5

Figure 4.52 Variation of drying shrinkage value for different mixes produced with AL having 14M SH solution and AL/FA=0.6

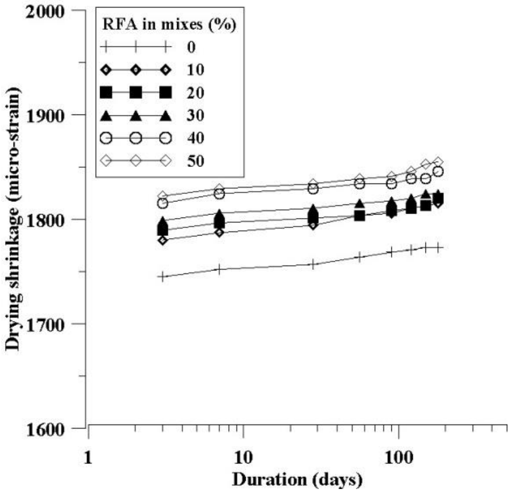
SS/SH = 1.0



SS/SH = 1.5



SS/SH = 2.0



SS/SH = 2.5

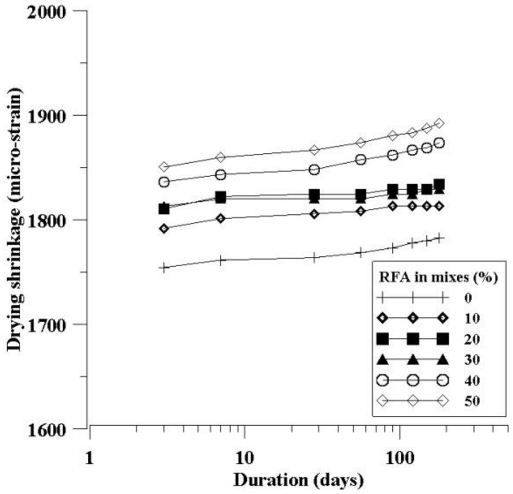
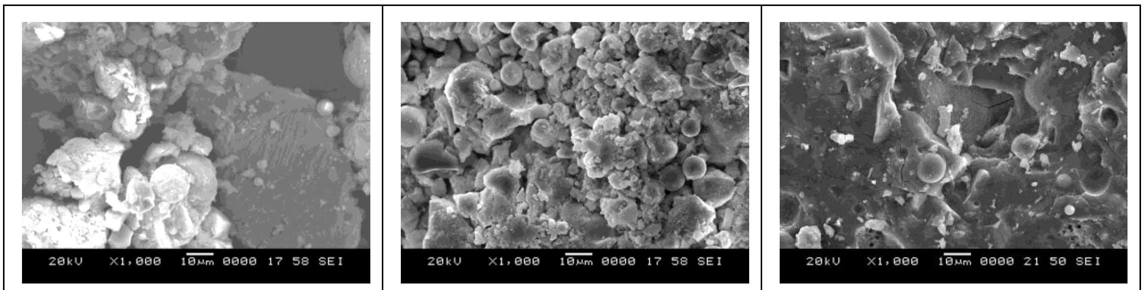


Figure 4.53 Variation of drying shrinkage value for different mixes produced with AL having 16M SH solution and AL/FA=0.6

4.6 MICROSTRUCTURE OF GEOPOLYMER MORTAR MIXES

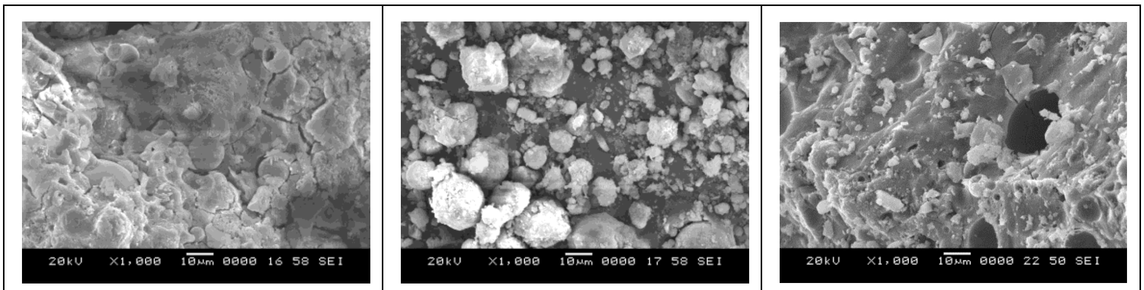
Samples were taken from the failure surface of the tested cubes for the study of microstructures of FA based geopolymer mortar mixes for which 28 days compressive strength was observed highest. For both the AL/FA ratios, geopolymer mortar mixes produced with 20% RFA were observed to gain the highest strength under all the curing regimes. Figure 4.54 (a, b& c) represents the SEM images of the samples obtained for the geopolymer mortar mixes produced with AL/FA = 0.4 & 20% RFA for ambient temperature curing, water curing and heat curing regime respectively. From the images, the presence of un-reacted fly ash particles (rounded one) is observed which leads to showing lesser strength than ambient temperature and water cured samples than that of heat cured samples. Chindaprasirt et al. (2018) also observed a dense matrix with few FA particles that were non-reacted, partially reacted, or both, in SEM images taken from the samples of alkali-activated FA having calcium-rich compounds.



(a) Ambient temp. cured (b) Water cured (c) Heat cured

Figure 4.54 SEM images of the samples taken from geopolymer mortar mixes produced with AL/FA=0.4 & 20% RFA

Figure 4.55 (a, b& c) represents the SEM images of the samples obtained for the geopolymer mortar mixes produced with AL/FA = 0.6 & 20% RFA for all the curing regimes. From the SEM images, it is clearly evident that geopolymer mortar mixes prepared with higher AL/FA ratio consists of more dense and uniform structures. Almost similar types of morphology have been observed for the all other mixes.



(a) Air cured (b) Water cured (c) Heat cured

Figure 4.55 SEM images of the samples taken from geopolymer mortar mixes produced with AL/FA=0.6 & 20% RFA

4.7 ENHANCEMENT OF THE PROPERTIES OF FLYASH BASED GEOPOLYMER

As difficulties were observed in the laboratory with respect to setting time of the fly ash based geopolymer binder, the present investigation also concentrated to address the drawbacks related to setting time. To enhance the properties of fly ash based geopolymer paste, experimental investigations had been conducted on the fly ash based geopolymer paste produced with different combination of SH solution and SS solution. Higher setting time is one of the barriers to use FA based geopolymer effectively in the construction industry. Although most of the researchers concluded that FA based geopolymer paste will show less duration for setting and high strength with heat curing. But heat curing is also not acceptable since practically it is difficult in many cases. Therefore, an experimental investigation was carried forward to improvise the setting and compressive strength of FA based geopolymer by introducing ground granulated blast furnace slag in the mixes. According to the guidelines given by Indian Standards for OPC (IS: 4031) standard consistency, initial and final setting time and compressive strength of geopolymer paste with 100% fly ash was determined and noted. Then, GGBS was incorporated at certain percentage levels to enhance the properties of fly ash geopolymer paste, and all the properties were determined.

4.7.1 Setting Time

To determine the setting time of FA geopolymer paste, the quantity of AL was considered to prepare the sample, based on the standard consistency value of the respective mixes. It has been observed that the initial and final setting time of geopolymer paste both reduced with the increment of the ratio of SS solution to SH. Initial setting time of geopolymer paste was found to be in the range of 260 minutes to 480 minutes and final setting time to be in the range of 1000 minutes to 1600 minutes in this study. Therefore, with respect to the initial and final setting time of OPC paste, FA based geopolymer paste requires more time for initial set and final set. Most of the researchers concluded that geopolymer paste will show less duration for setting and high strength with the heat curing. The problems regarding setting time of FA based geopolymer paste were addressed later without adopting heat curing.

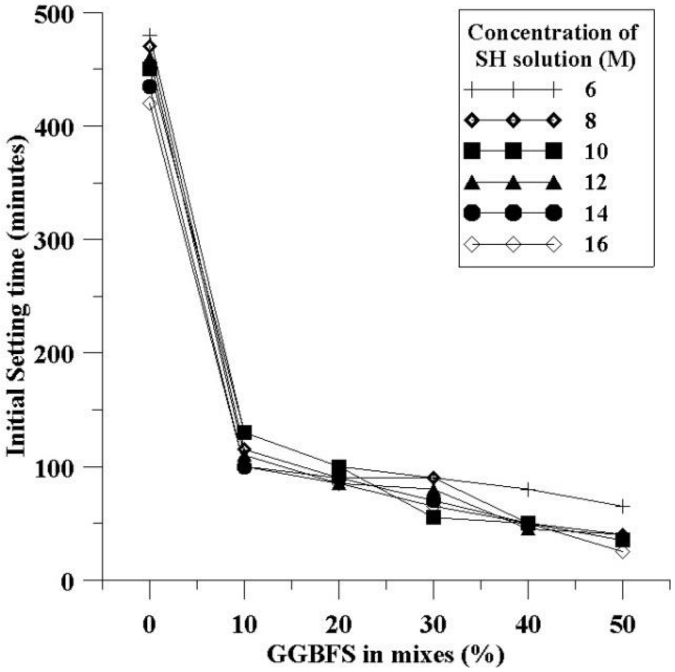


Figure 4.56 Initial Setting Time of Geopolymer Paste

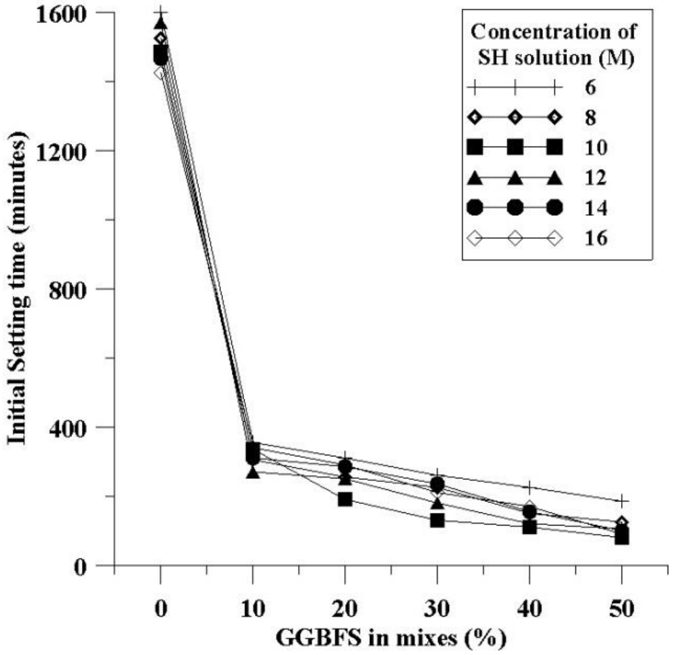


Figure 4.57 Final Setting Time of Geopolymer Paste

It has been observed from this experimental investigation that initial and final setting time of geopolymer paste both reduced significantly with the increment of the GGBFS in the mixes. In this study, very high initial and final setting time of geopolymer paste made with FA only was found. Incorporation of GGBFS in mixes reduces the range of initial setting time from 420 - 480 minutes to 25-130 minutes and final setting time from 1425-1600 minutes to 90- 355 minutes. So, problems regarding setting time of FA based geopolymer can be solved by the incorporating GGBFS in mixes. Initial setting time of geopolymer paste was found to be reduced by 73%- 94% and final setting time was reduced by 77% - 92% with the addition of GGBFS from 10% to 50% in the mixes when compared to the setting time of geopolymer paste mixes without GGBFS. The percentage of reduction in setting time is high for the mixes having higher quantity of GGBFS. Figure 4.59 and Figure 4.60 shows the variation of initial setting time (IST) and the final setting time (FST) of the geopolymer paste with different percentage of GGBFS respectively. Setting of OPC is directly related with the development of calcium silicate hydrate gel (C-S-H) by the reaction between cement and water. Similarly, geopolymer paste is typically dependent on the development of silico-aluminate gel. As the quantity of CaO is more in the GGBFS, geopolymer paste mixes produced with GGBFS may form C-S-H gel also along with silico-aluminate gel at the early duration. As a result, geopolymer paste produced with high quantity of GGBFS shows appreciable less time for initial setting and final setting.

4.7.2 Compressive Strength

Compressive strength of geopolymer paste (cube samples cured at ambient temperature) was found to be increasing trend while the concentration of SH solution and the percentage of GGBFS in mixes both were increased. Compressive strength of geopolymer paste at 7 days, 28 days and 56 days were increased by 70% - 75%, 50% - 61% and 50% - 61% respectively with the addition of GGBFS in the mixes when compared to the compressive strength of geopolymer paste mixes without GGBFS. Increase in compressive strength was observed more when the content of GGBFS in mixes was higher.

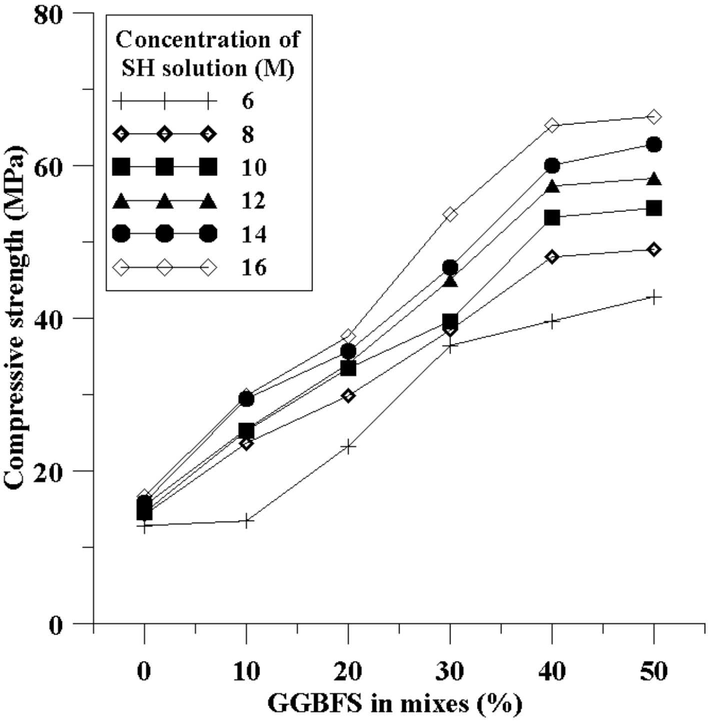


Figure 4.58 Compressive strength of geopolymer paste at 7 days

Compressive strength of geopolymer paste is attributed by generating of silico-aluminate gel and poly-condensation to form 3-D network of silico-aluminates structures. Higher concentration of SH solutions helps to form sufficient silico-aluminate gel and leads to poly-condensation. As a result, stable 3-D network of silico-aluminate structures is produced to provide higher compressive strength to the FA based geopolymer. Apart from 3-D network of silico-aluminate structure, there may be the formation of C-S-H gel as CaO content in GGBFS is high. Formation of C-S-H gel along with silico-aluminate structures in the paste mixes also contribute in order to gain high strength significantly.

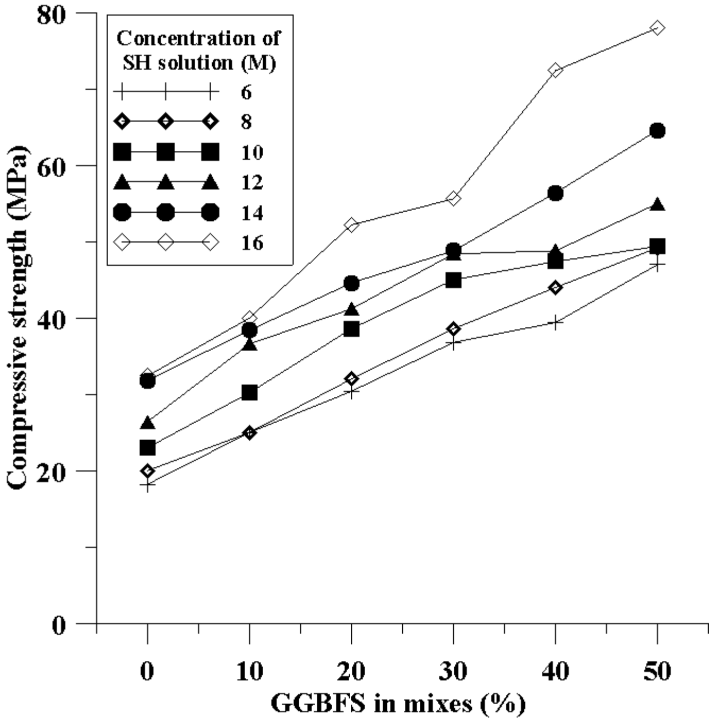


Figure 4.59 Compressive strength of geopolymer paste at 28 days

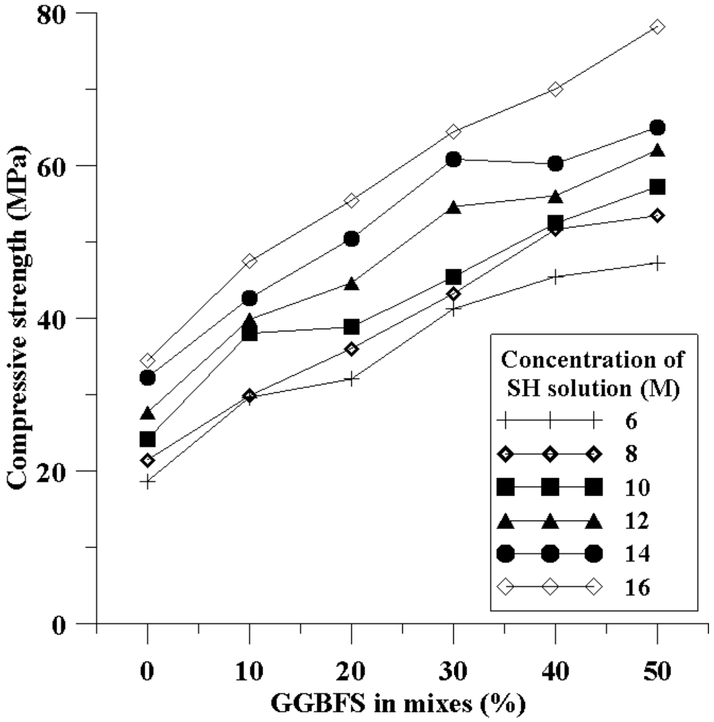
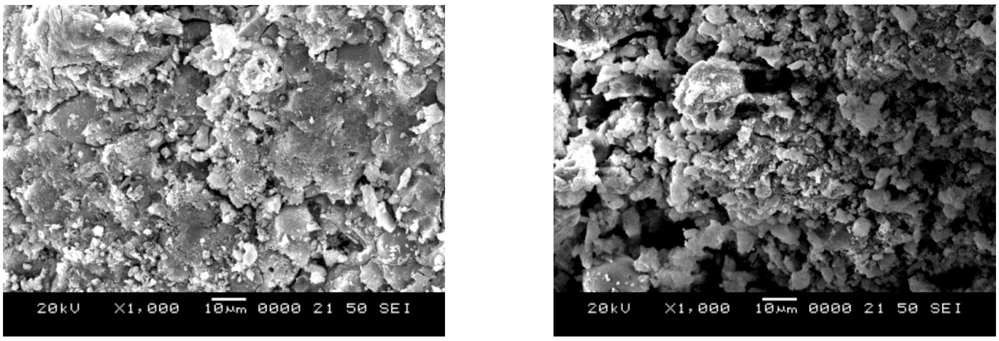


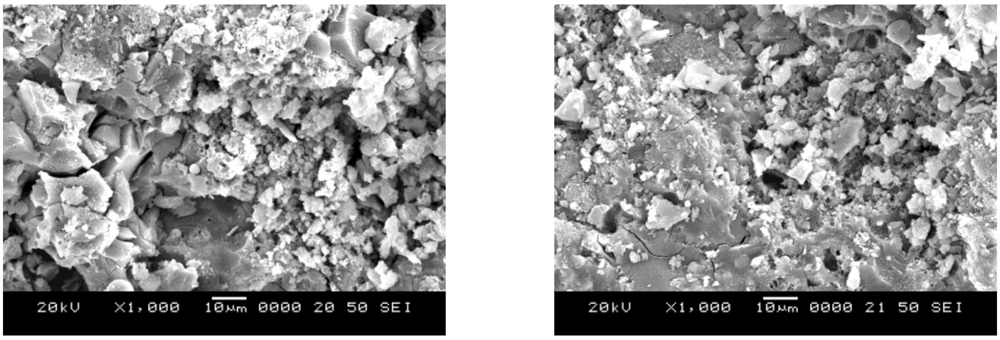
Figure 4.60 Compressive strength of geopolymer paste at 56 days

The enhancement profile of compressive strength of geopolymer paste with different percentage of GGBFS at 7 days, 28 days and 56 days are shown in the Figure 4.61,

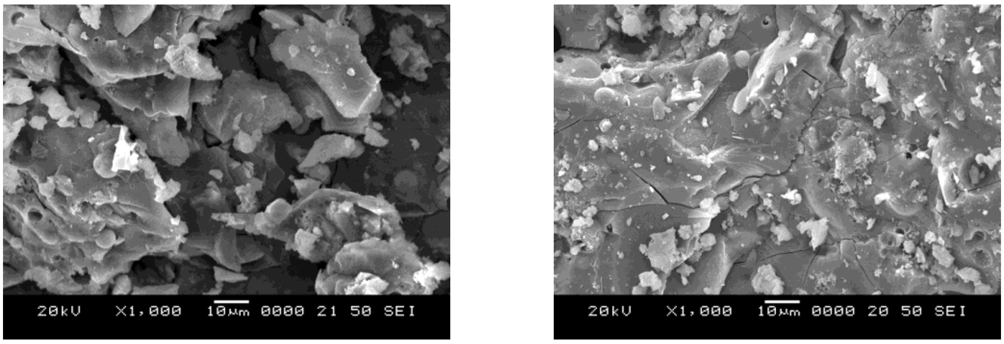
Figure 4.62 and Figure 4.63 respectively. Highest compressive strength of geopolymer paste at 7 days, 28 days and 56 days were observed as 66.4 MPa, 78 MPa, and 78.2 MPa respectively for the mix with 16M SH solution and 50% GGBFS. Gaining of compressive strength of geopolymer paste mixes after 28 days is observed to be very less. The mix with 16M SH solution and 50% GGBFS, which exhibited the highest strength, has 14.8 % gain in compressive strength after 7 days and 0.26% gain after 28 days.



(a) mix with 0 % GGBFS **(b) the mix with 10 % GGBFS**



(c) mix with 20 % GGBFS **(d) mix with 30 % GGBFS**



(e) mix with 40 % GGBFS **(f) mix with 50 % GGBFS**

Figure 4.61 SEM Images of samples taken from different mixes

Samples for the analysis of the microstructure of the geopolymer paste with different dosage of GGBFS were taken from the failure surfaces of the cube samples, which had shown the highest strength in this experimental study. From the SEM images [Figure 4.61(a-f)], it can be concluded that higher replacement of FA by GGBFS in the mixes helps to form denser structure. As a result, higher strength was observed for the geopolymer paste with the higher quantity of GGBFS. The presence of calcium silicate hydrate gel becomes prominent with the increment of the quantity of GGBFS in the mixes and as a result, more dense structures were formed.

4.8 CLOSURE

This chapter interprets the observation of different tests to determine workability, water absorption capacity, compressive strength, and drying shrinkage of fly ash geopolymer mortar mixes with recycled fine aggregate as partial replacement of natural fine aggregate (sand). Additional investigation has been conducted to enhance the properties of fly ash based geopolymer paste by incorporating GGBFS as partial replacement of fly ash. The results indicate that recycled fine aggregate can be used as fine aggregate effectively to produce eco-friendly geopolymer mortar mixes with the desired properties.

CHAPTER 5

PREDICTION MODEL USING ANN

5.1 INTRODUCTION

Prediction models using the artificial neural network (ANN) are being established with the help of the observed results in the laboratory. All those models to predict the properties of the fly ash based geopolymer mortar mixes are discussed in details in this chapter.

5.2 ARTIFICIAL NEURAL NETWORK (ANN)

Artificial Neural Network (ANN) is information processing unitsof artificial neurons, which is basically inspired by the way biological nerve units such as the human brain; perform a particular task or function of interest. Artificial neural networks (ANNs) are flexible computing frameworks that resemble the structure of a nervous system.ANN represents highly ideological mathematical models of our present understanding of complex systems. ANN models have the ability to learn and generalize the problems even when input data contain error or incomplete.

ANNs have been started to use extensively in the field of civil engineering. These networks operate on the principle of learning from a training set. Neural networks are typically organized in layers, which consists of number of interconnected ‘nodes’ containing an ‘activation function’. In general, the advantages of ANNs over other statistical models are as followings.

- The application of ANNs has the ability to identify the complex non-linear relationship between input and output data sets without the understanding of the phenomena.
- ANNs have non-linear properties because neurons activate a non-linear filter called activation function.
- Multiple input parameters having different characteristics enable ANNs to indicate the time-space variability, and

- ANNs have the quality to adjust any changes in the problem environments (Kim and Valdes, 2003).

The efficiency of an artificial neuron network depends on the learning ability of neurons, which is usually obtained by assigning weights by the chosen algorithm. Figure 5.1 represents the basic architecture of ANN with input, hidden layers, and outputs, which are connected by arrows.

The predicted value or output from an artificial neuron network (P) is given by the following relationship:

$$P = f(X) = f\left(\sum_{j=1}^{n} W_j\, I_j\right) \qquad \ldots(5.1)$$

where W_j is the weight assigned to the input parameters, I_j is the input parameters and the function f(X) is activation(transfer) function.

The variable X is defined as a scalar product of weight and input vectors;

$$X = W_1I_1 + W_2I_2 + W_3I_3 + \ldots + W_nI_n \qquad \ldots(5.2)$$

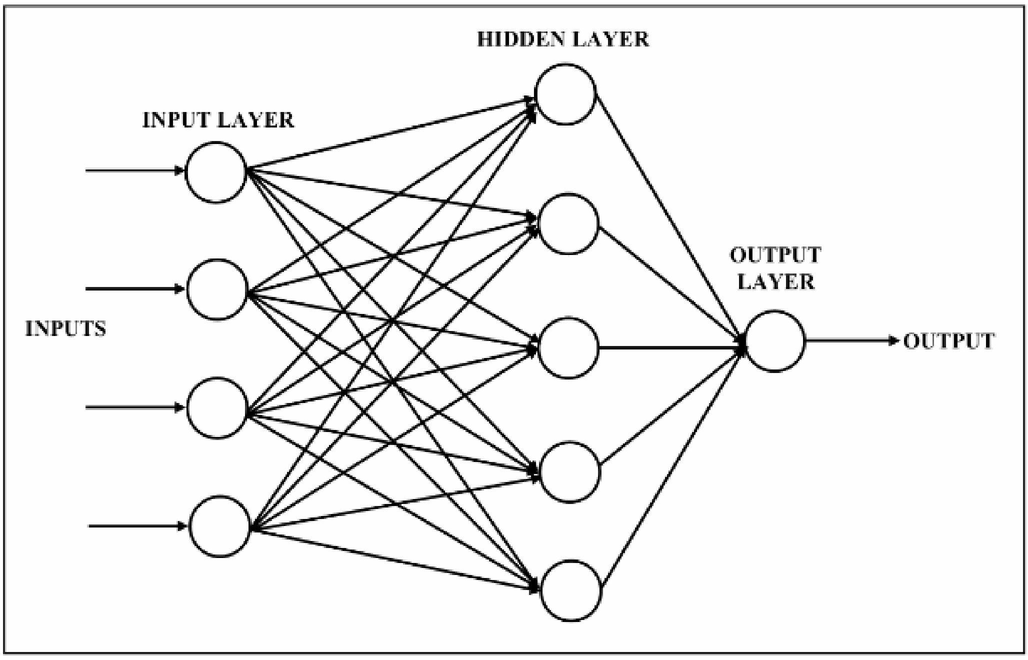


Figure 5.1 Architecture of ANN

Application of Artificial Neural Networking in various civil engineering problems has increased in recent days. ANN have been started to be used for recognizing complicated patterns and find solutions to problems, which are too complex to be modeled accurately by traditional computing methods. This modeling approach is very useful as it learn from examples since it is often easy to learn if we have a set of experimental results rather than theoretical guesses. Traditional computing solutions are based on predefined rules or equations, which gave a clear definition of the problem. Artificial Neural Network (ANN) works in reducing such lengthy algorithms without satisfying in results.

5.3 PAST STUDIES ON PREDICTION OF PROPERTIES OF MORTAR/CONCRETE MIXES

Mashadban et al. (2016) conducted an experimental study to determine fresh, mechanical and durability properties of self-compacting concrete (SCC) mixes. Polyphenylene sulfide fibres of 0.1, 0.2, 0.3 and 0.4% (by volume); steel fibres of 0.1, 0.2, 0.3 and 0.4% (by volume) and unreinforced were used to produce total 9 SCC concrete mixes in this study. After that, the obtained data from experiments were used to generate a model for forecasting the properties of different SCC concrete mixes using ANN and particle swarm optimization algorithm (PSOA). They found that PSOA integrated with the ANN predicted the properties of fiber reinforced SCC concrete mixes with a high level of accuracy.

Yaman et al. (2017) predicted the performance of ANN in proportioning of ingredients for SCC mixes considering 28 - day compressive strength and slump values as input parameters. All the data used in this study were collected from the literature. Multi input - multi output ANN model and multi input - single output ANN model was used to get the best prediction model for the SCC ingredients. Cement content, fly ash, water/binder ratio, fine aggregate, coarse aggregate, and superplasticizers were considered as outputs for the developed model. Themulti input – single output ANN prediction model where the six outputs are predicted separately exhibited higher accuracy level than that of multi input - multi output ANN prediction model.

Khashman and Akpinar (2017) investigatedthe application of ANNs to predict and categorize the different concrete mixes based on compressive strengthas the mixes of low, moderate and high strength. Water content, fine aggregates, cement content, coarse aggregates, fly ash, superplasticizers, slag, andage were considered as input parameters to develop the ANN model for forecasting compressive strength of different concrete mixes. Results of this study showed high efficiency for categorizing the compressive strength as low, moderate and high.

Eskandari-Naddaf et al. (2017) used ANN to predict the compressive strength of different mortar mixes. Mortar mixes were produced with different types of cement having the strength of 32.5, 42.5, and 52.5 MPa. For generating data set, 54 mortar mixes were designed and obtained the results of 810 specimens with the consideration of 6 water-cement ratios (W/C) (0.25, 0.3, 0.35, 0.4, 0.45, and 0.5) and 3 sand/cement ratios (S/C) (2.5, 2.75, and 3) along with the different types of cement. An effort was there to develop ANN prediction model with and without the consideration of the strength class of cement as an input parameter. High level of accuracy of the generated ANN prediction model for compressive strength of different mortar mixes was achieved when the different types of cement with respect to strength was considered as an input parameter.

Naderpour et al. (2018) developed model using ANN to predict compressive strength of recycled aggregate concrete. 139 data collected from 14 published literature were used to develop the ANN prediction model. Water-cement ratio, water absorption capacity, fine aggregate, natural coarse aggregate, recycled coarse aggregate, water-total material ratio were considered as input parameters to predict the compressive strength of the concrete. From their study, they concluded that ANN can be used as an efficient tool to forecast the compressive strength of recycled aggregate concrete.

Jin et al. (2018) developed modelto predict the properties of eco-friendly concrete mixes having alternative or waste materials using non-linear and mixed regression analysis. Higher level of accuracy in prediction was observed for the model developed using non-linear and mixed regression than the prediction model established using the linear method.

Paul et al. (2018) conducted experimental study anddeveloped prediction model using ANN for forecasting the mechanical strength of recycled aggregate concrete where the recycled coarse aggregate replacement level, water/cement ratio, aggregate/cement ratio, air content in the concrete mixes were considered as input parameters. Predicted values by ANN model for compressive and splitting tensile strength of concrete mixes were observed as almost similar to the experimental values.

Shi et al. (2018) developed model to predict the mechanical and electrical characteristics of engineered cementitious composites using the artificial neural network (ANN) technique. For training of the developed ANN model, data were collected from published literature related to engineered cementitious composites with polyvinyl alcohol fiber or steel fiber. To check the performances of the developed ANN prediction models, experimental investigations were also conducted for engineered cementitious composites of various compositions. Highly correlation between the predicted values from the ANN model and experimental observations was observed.

5.4 CONSIDERATION FOR DEVELOPING ANN MODEL

Therefore, it has been seen from the literature that the ANN technique can be used as one the most effective technique to predict the properties of mortar/concrete mixes. In this study, obtained data from the experimental investigations, which was explained in the previous chapter, has been considered for the development of the prediction model using ANN. Individual models using ANN have been developed to predict water absorption capacity, compressive strength at different durations and drying shrinkage at 180 days of different fly ash based geopolymer mortar mixes. Concentration of SH Solution in AL, SS/SH ratio in AL, RFA content (%) and AL/FA ratio have been considered as input parameters to predict water absorption capacity of the produced fly ash based geopolymer mortar mixes. To develop the ANN prediction model for compressive strength and drying shrinkage properties of fly ash based geopolymer mortar mixes, curing regime has been also considered as input parameter along with earlier mentioned parameters.

All the ANN models had been developed with the MATLAB (R2015a) ANN toolbox. The tansig, purelin, and logsig transfer functionshave been considered as the activation functions for the hidden layer and a linear function is considered as the activation function for the output layer. The number of neurons in hidden layers also has been varied from (2n – 2) to (2n + 2), where n is the total number of inputs. Performances of the developed ANN prediction models have been evaluated by the different statistical indices explained in a subsequent section in details.

5.5 PERFORMANCE EVALUATION USING STATISTICAL INDICES

The performance of predictions of all the models for each property of fly ash based geopolymer mortar mixes has been evaluated on the basis of different statistical indices. The statistical indices tell us about the confidence level one can have on the prediction of the model. Correlation coefficient (R), coefficient of determination (R^2-value), Root mean square error (RMSE), Mean absolute error (MAE), Mean absolute percentage error (MAPE) and Accuracy performance (AP) have been used as statistical indices to assess the performances of all the developed models. The R^2 determines the degree of linear correlation between the predicted values and the observed values. RMSE provides the variant of the total errors, while the MAE, MAPE provide the absolute error information. Lower RMSE, MAE and MAPE values indicate that the performance of the prediction model is better and R values close to 1 indicate better performance of the model.

Coefficient of determination (R^2)

$$\mathbf{R^2 = 1 - \frac{\sum_{i=1}^{N}(E_i - \hat{P}_i)^2}{\sum_{i=1}^{N}(E_i - \bar{E}_i)^2}} \qquad \text{...(5.3)}$$

Where,

$$\mathbf{\bar{E}_i = \frac{1}{N}\sum_{i=1}^{N} E_i} \qquad \text{...(5.4)}$$

$\bar{E}_i$ = mean value taken over N

E_i = observed value from experiments

$\hat{P}_i$ = predicted value and

N = number of data

Root Mean Square Error (RMSE)

$$SSE = \sum_{i=1}^{N}(\mathrm{E_i} - \mathrm{\widehat{P}_i})^2 \qquad ...(5.5)$$

$$RMSE = \sqrt{\frac{SSE}{N}} \qquad ...(5.6)$$

where SSE = sum of the squared errors

N = number of data used

Mean Absolute Error (MAE)

$$\mathrm{MAE} = \frac{1}{N}\sum_{i=1}^{N}\left|E_i - \widehat{P}_i\right| \qquad ... (5.7)$$

where,

$\overline{\mathrm{E}}_\mathrm{i}$ = mean value taken over N

$\mathrm{E_i}$ = observed value from experiments

$\mathrm{\widehat{P}_i}$ = predicted value and

N = number of data

Mean Absolute Percentage Error (MAPE)

$$\mathrm{MAPE} = \frac{1}{N}\frac{\sum_{i=1}^{N}\left|E_i - \widehat{P}_i\right|}{E_i} \qquad ... (5.8)$$

Accuracy Performance (AP)

$$\mathrm{AP} = (100 - MAPE) \qquad ...(5.9)$$

5.6 PREDICTION MODEL FOR WATER ABSORPTION CAPACITY

Using different transfer function and different number of hidden neurons, total 15 ANN models have been developed to predict the water absorption capacity of fly ash based geopolymer mortar mixes. Concentration of SH Solution in AL, SS/SH ratio in AL, RFA content (%) and AL/FA ratio have been considered as input parameters for developing the ANN models. Table 5.1 represents the performances of all the developed prediction models with respect to different statistical indices. From the analysis, it has been observed that all developed ANN models have shown high level of accuracy. ANN model 4 - 9 - 1 exhibits high level of accuracy and can be concluded as the best model to predict the water absorption capacity of the different mixes. Figure 5.2, Figure 5.3 and Figure 5.4 represent the regression plot, training state and performance of the observed best prediction ANN model for water absorption capacity of different geopolymer mortar mixes containing RFA partially.

Table 5.1 Performance of developed ANN model for water absorption capacity

Network details	**transfer function**	**R-value**	**R^2**	**RMSE**	**MAE**	**MAPE**	**AP**
4 - 6 - 1	tansig	0.995	0.990	0.028	0.020	9.726	90.274
4 - 7 - 1		0.995	0.990	0.030	0.022	10.436	89.564
4 - 8 - 1		0.994	0.988	0.033	0.022	10.398	89.602
4 - 9 - 1		**0.997**	**0.994**	**0.025**	**0.017**	**8.103**	**91.897**
4 - 10 - 1		0.995	0.990	0.029	0.019	8.469	91.531
4 - 6 - 1	purelin	0.985	0.970	0.053	0.034	13.030	86.970
4 - 7 - 1		0.986	0.972	0.050	0.033	12.130	87.870
4 - 8 - 1		0.985	0.970	0.051	0.034	12.655	87.345
4 - 9 - 1		0.986	0.972	0.050	0.034	12.532	87.468
4 - 10 - 1		0.986	0.972	0.050	0.033	12.436	87.564
4 - 6 - 1	logsig	0.993	0.986	0.035	0.024	10.631	89.369
4 - 7 - 1		0.996	0.992	0.027	0.019	8.292	91.708
4 - 8 - 1		0.995	0.990	0.030	0.021	9.390	90.610
4 - 9 - 1		0.996	0.992	0.028	0.019	9.516	90.484
4 - 10 - 1		0.995	0.990	0.028	0.019	8.718	91.282

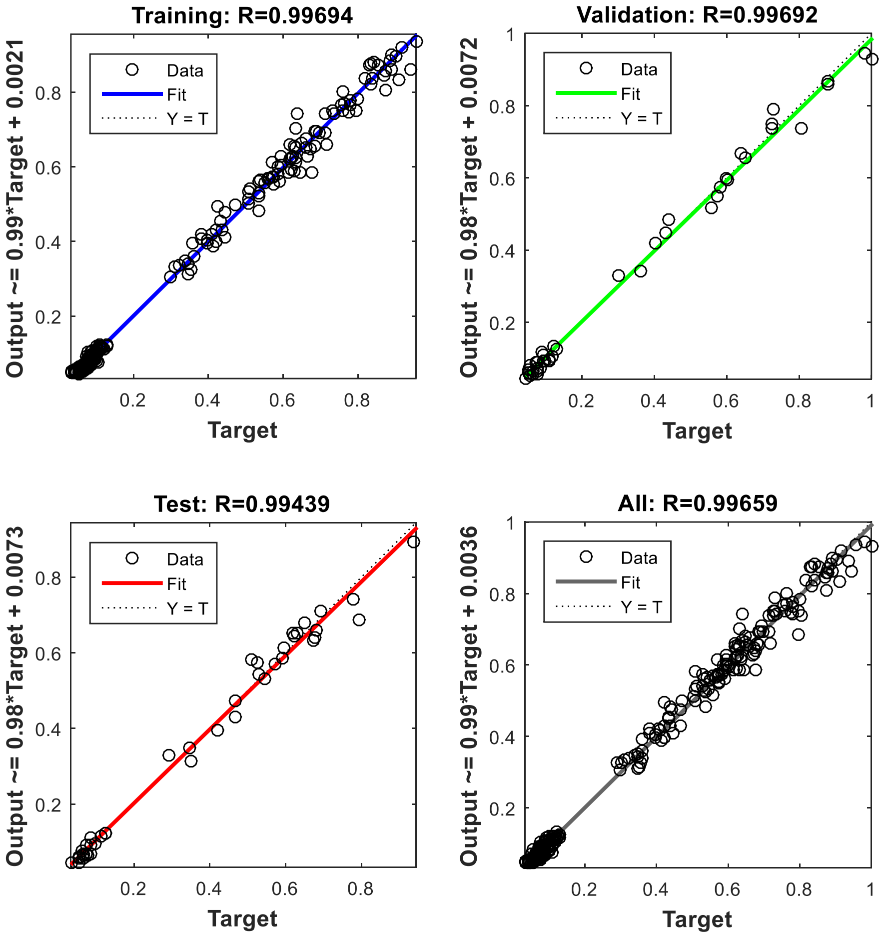


Figure 5.2 Regression plot of best ANN model (4-9-1) to predict water absorption capacity of mixes

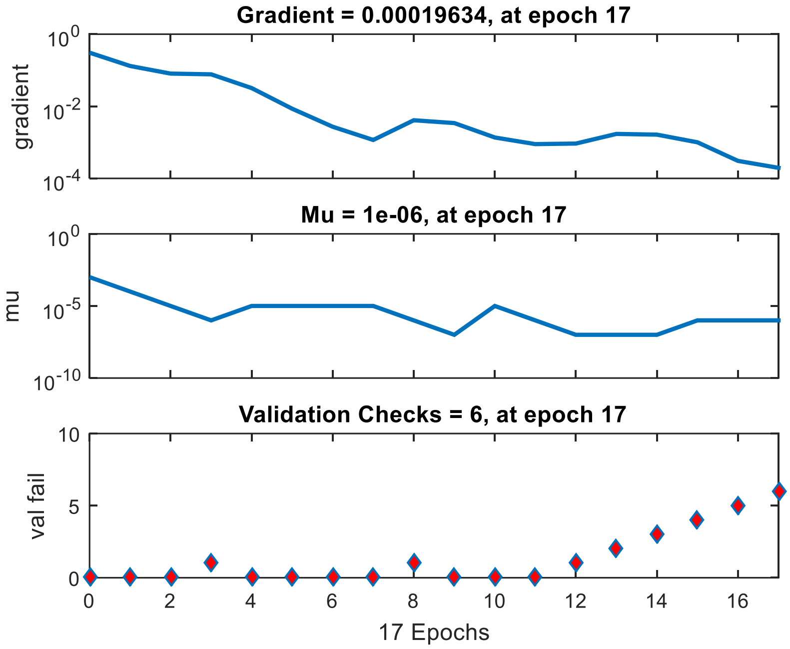


Figure 5.3 Training state of best ANN model (4-9-1) to predict water absorption capacity of mixes

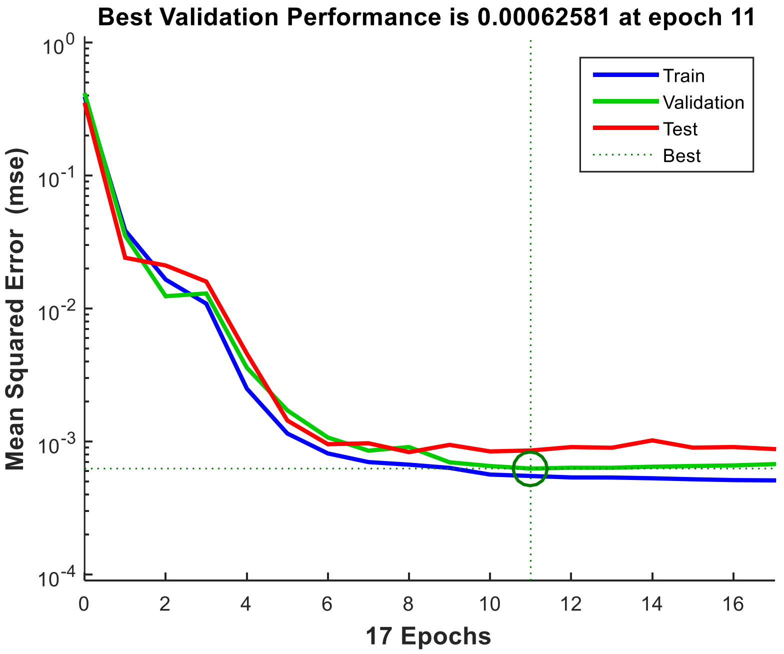


Figure 5.4 Performance plot of best ANN model (4-9-1) to predict water absorption capacity of mixes

5.7 PREDICTION MODEL FOR COMPRESSIVE STRENGTH

Concentration of SH Solution in AL, SS/SH ratio in AL, RFA content (%), AL/FA ratio and curing regimes have been considered as input parameters for developing the ANN models for forecasting compressive strength at 3 days, 7 days, 28 days and 56 days. The performances of all the developed ANN prediction models with respect to different statistical indices are presented in Table 5.2. From the analysis, it has been observed that ANN models have shown high accuracy level except the ANN models developed using purelin transfer function. ANN model 5-12-4 exhibits high level of accuracy and can be concluded as the best model to predict the water absorption capacity of the different mixes. Figure 5.5, Figure 5.6 and Figure 5.7 represent the regression plot, training state and performance of the observed best prediction ANN model for compressive strength at different ages of different geopolymer mortar mixes containing RFA partially.

Table 5.2 Performance of developed ANN model for compressive strength

Network details	**transfer function**	**R-value**	**R^2**	**RMSE**	**MAE**	**MAPE**	**AP**
5 - 8 - 4	tansig	0.946	0.895	0.065	0.046	11.662	88.338
5 - 9 - 4		0.953	0.908	0.060	0.044	11.376	88.624
5 - 10 - 4		0.959	0.920	0.056	0.041	10.595	89.405
5 - 11 - 4		0.958	0.918	0.057	0.042	10.659	89.341
5 - 12 - 4		0.961	0.924	0.055	0.041	10.277	89.723
5 - 8 - 4	purelin	0.698	0.487	0.141	0.111	31.361	68.639
5 - 9 - 4		0.698	0.487	0.141	0.110	30.459	69.541
5 - 10 - 4		0.698	0.487	0.141	0.110	31.148	68.852
5 - 11 - 4		0.698	0.487	0.141	0.111	31.650	68.350
5 - 12 - 4		0.698	0.487	0.141	0.110	31.055	68.945
5 - 8 - 4	logsig	0.947	0.897	0.064	0.047	12.275	87.725
5 - 9 - 4		0.957	0.916	0.058	0.042	10..607	89.393
5 - 10 - 4		0.952	0.906	0.061	0.043	10.893	89.107
5 - 11 - 4		0.947	0.897	0.064	0.048	11.954	88.046
5 - 12 - 4		**0.961**	**0.924**	**0.055**	**0.040**	**10.154**	**89.846**

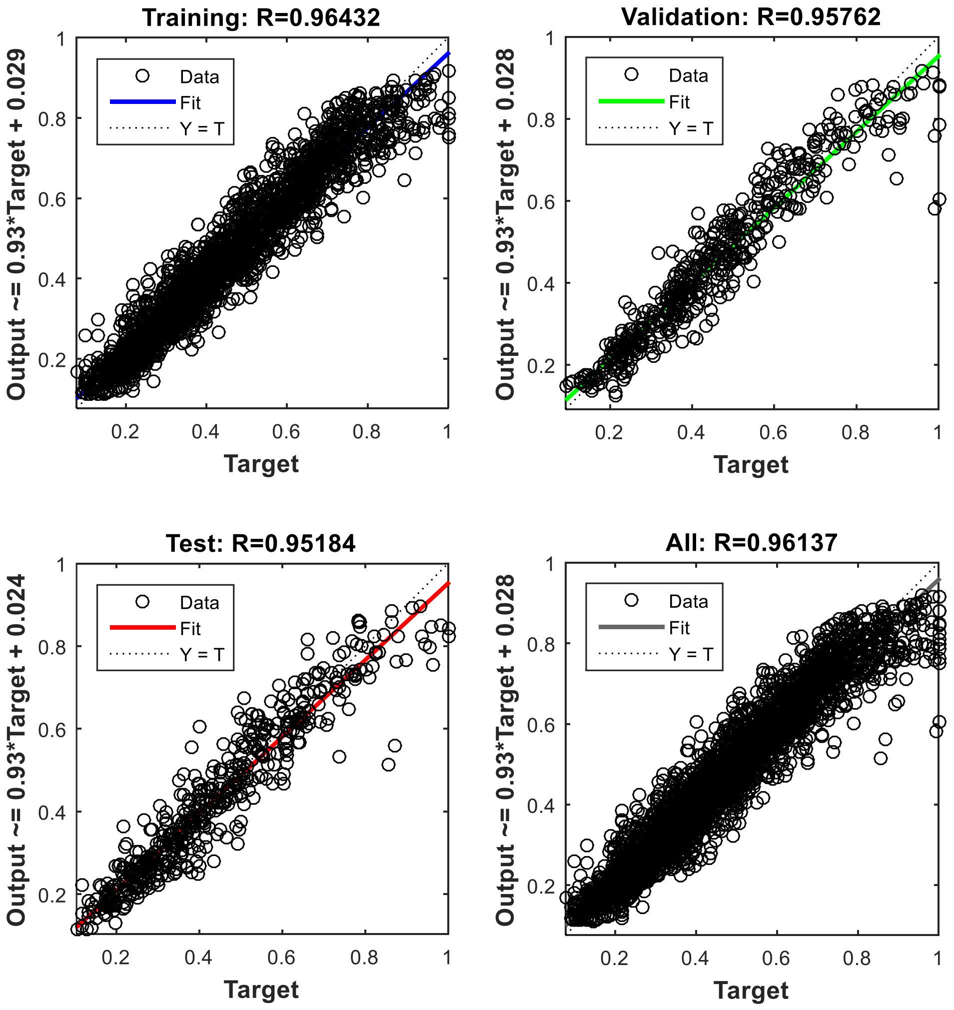


Figure 5.5 Regression plot of best ANN model (5-12-4) to predict compressive strength of mixes

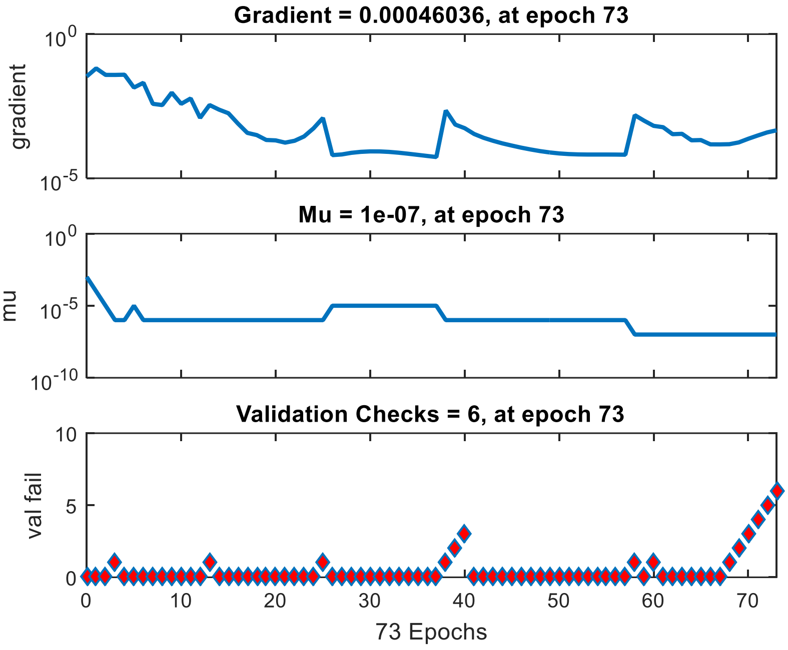


Figure 5.6 Training state of best ANN model (5-12-4) to predict compressive strength of mixes

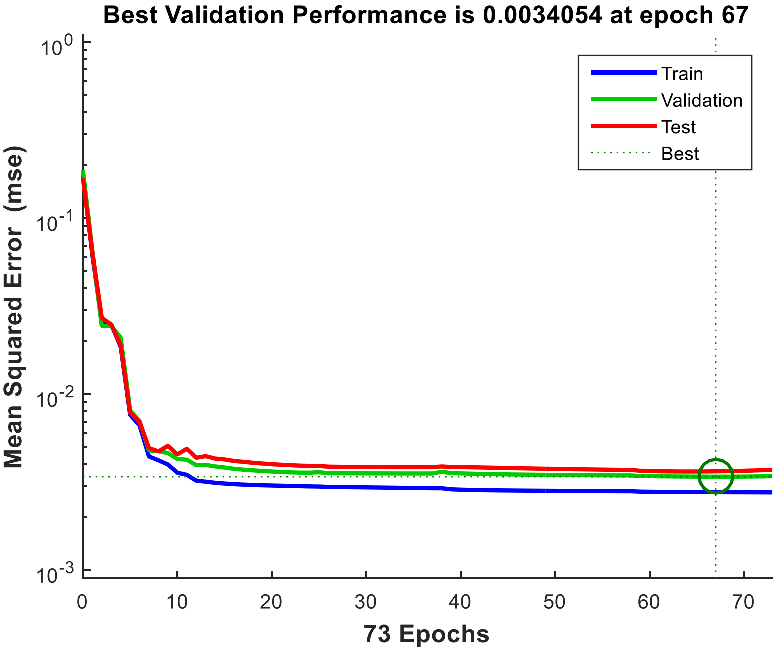


Figure 5.7 Performance plot of best ANN model (5-12-4) to predict compressive strength of mixes

5.8 PREDICTION MODEL FOR DRYING SHRINKAGE

Table 5.3 Performance of developed ANN model for drying shrinkage

Network details	**transfer function**	**R-value**	**R^2**	**RMSE**	**MAE**	**MAPE**	**AP**
5 - 8-1	**tansig**	0.976	0.953	0.023	0.016	2.296	97.704
5 - 9 - 1		0.98	0.960	0.021	0.015	2.148	97.852
5 - 10 - 1		0.984	0.968	0.019	0.014	1.899	98.101
5 - 11 - 1		0.984	0.968	0.019	0.012	1.729	98.271
5 - 12 - 1		**0.988**	**0.976**	**0.017**	**0.012**	**1.643**	**98.357**
5 - 8 - 1	purelin	0.946	0.895	0.035	0.027	3.854	96.146
5 - 9 - 1		0.946	0.895	0.034	0.027	3.798	96.202
5 - 10 - 1		0.946	0.895	0.035	0.027	7.678	92.322
5 - 11 - 1		0.946	0.895	0.035	0.027	3.829	96.171
5 - 12 - 1		0.946	0.895	0.035	0.027	3.864	96.136
5 - 8 - 1	logsig	0.959	0.920	0.030	0.022	3.042	96.958
5 - 9 - 1		0.983	0.966	0.020	0.014	1.960	98.040
5 - 10 - 1		0.96	0.922	0.030	0.021	2.990	97.010
5 - 11 - 1		0.988	0.976	0.017	0.012	1.670	98.330
5 - 12 - 1		0.987	0.974	0.017	0.012	1.689	98.311

To predict the drying shrinkage value of different geopolymer mortar mixes using ANN, concentration of SH Solution in AL, SS/SH ratio in AL, RFA content (%), AL/FA ratio and curing regimes have been considered as input parameters. The performances of all the developed ANN prediction models with respect to different statistical indices are presented in Table 5.3. From the analysis, it has been observed that all the ANN models have predicted drying shrinkage almost similar. ANN model 5-12-1 exhibits high level of accuracy and can be concluded as the best model to predict the water absorption capacity of the different mixes. Figure 5.8, Figure 5.9 and Figure 5.10 represent the regression plot, training state and performance of the observed best prediction ANN model for compressive strength at different ages of different geopolymer mortar mixes containing RFA partially.

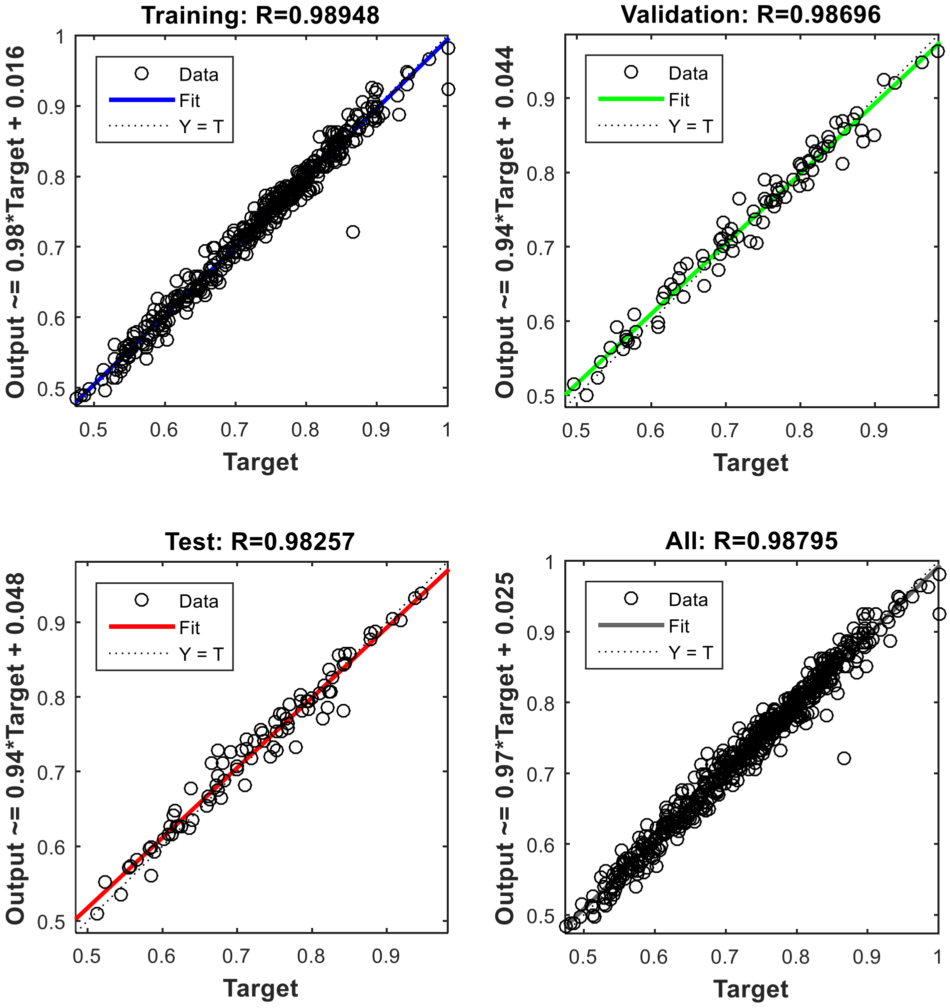


Figure 5.8 Regression plot of best ANN model (5-12-1) to predict drying shrinkage of mixes

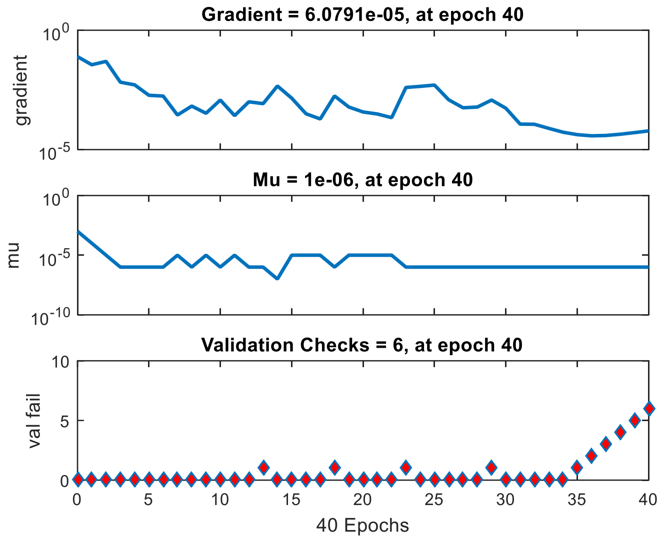


Figure 5.9 Training state of best ANN model (5-12-1) to predict drying shrinkage of mixes

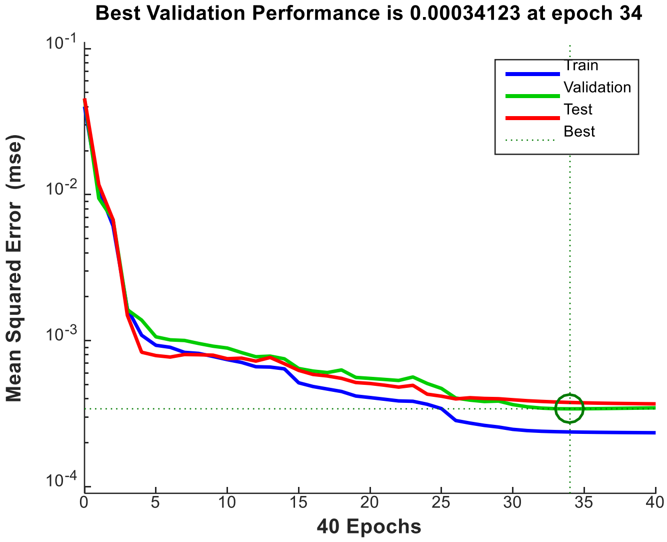


Figure 5.10 Performance plot of best ANN model (5-12-1) to predict drying shrinkage of mixes

5.9 CLOSURE

Prediction models using ANN technique have been developed to forecast the properties of different fly ash based geopolymer mortar mixes. Data has been taken from the experimental work explained in the earlier chapter. From the observations, it can be said that the ANN technique is one of the best suitable approaches to predict the properties of the mortar mixes with a high precision level.

Chapter 6

CONCLUSIONS

6.1 INTRODUCTION

The use of recycled materials generated from construction and demolition waste is growing across the world. Use of recycled aggregates in the construction activities is one of the most eco-friendly, responsible and feasible approaches of meeting the challenges of sustainability in the construction industry. The main objective of the present work is to investigate the effect of using recycled aggregatein lieu of natural aggregate on the properties of fly ash based geopolymer mortar mixes. As elaborated in the previous chapters, an experimental investigation on the effects of different parameters e.g. RFA content (%), concentration of SH solution in AL, SS/SH ratio in al, AL/FA ratio, different curing regimes on the properties (workability, water absorption, compressive strength and drying shrinkage) on fly ash based geopolymer mortar. The test results obtained are analyzed and discussed in the previous chapters. Based on the detailed analysis, the important findings are summarized in this chapter.

6.2 CONCLUSIONS ON CHARACTERISTICS OF RFA

- Quality of aggregates has an active contribution on the properties of mortar mixes. Water absorption capacity of RFA is significantly more than natural fine aggregates i.e. locally procured sand. It may cause problems of getting proper workable mortar mixes. In this study, it had been observed that the water absorption capacity of RFA is 9.2 times more than the water absorption capacity of locally procured sand.
- Gradation of RFA can be maintained as per the requirements for the mixes. Different size of particles of the produced RFA can be stored separately and mixed all the fractions to required percentage level according to the codal provisions or requirement before its use. In this study, generated RFA conformed zone I according to the guidelines given by IS: 383-2016, but all the combinations of RFA and NFA conformed to zone II.

6.3 CONCLUSIONS ON WORKABILITY OF GEOPOLYMER MORTAR MIXES

- Fly ash based geopolymer mortar mixes became stiffer condition when the RFA content in mixes was increased due to higher water absorption capacity of RFA. Mortar mixes having 50% RFA were observed as the stiffest mixes.
- High workable geopolymer mortar mixes having RFA partially can be produced with the consideration of the ratio of alkaline liquid to the binder as 0.8. But the setting time of mortar mixes with AL/FA ratio of 0.8 was very high. Therefore, de-molding of the cube samples becomes very difficult. With AL/FA=0.4, mortar mixes were stiffer mixes and during flow test, collapse occurred.
- There wasno significant variations found in the flow test when the concentration of SH solution and SS/SH ratio in AL werevaried.

6.4 CONCLUSIONS ON WATER ABSORPTION CAPACITY OF GEOPOLYMER MORTAR MIXES

- As the percentage level of NFA by RFA is higher, water absorption capacity ofgeopolymer mortar mixes were found to be higher. Higher water absorption capacity of RFA itself may be the reason for the higher water absorption of the RFA.
- Higher water absorption capacity of fly ash based geopolymer mortar mixes had been observed for the mixes produced with the higher concentration of SH solution.
- Significantly lower water absorption capacity of mortar mixes had been observed when AL/FA=0.6 was adopted.

6.5 CONCLUSIONS ON COMPRESSIVE STRENGTH OF GEOPOLYMER MORTAR MIXES

- Mortar mixes having 10% to 20% RFA content showed consistent better performance with respect to compressive strength of 0% RFA. Beyond 20% RFA content in mixes leads compressive strength in decreasing trend. But most of the mixes showed better strength with compared to the strength of mortar mixes having no RFA (control mixes).

- Most of the mixes exhibited higher compressive strength when mixes were produced with higher concentration of SH solution. When the SS/SH ratio was adopted as 1.5, then most of the mixes showed better performance. This combination of alkaline liquid helps to form silico-aluminate structures, which are mainly responsible for attaining strength by fly ash based geopolymer mortar.
- Higher compressive strength had been observed for the mixes produced with higher ratio of alkaline liquid to the binder. Mixes produced with AL/FA =0.6 showed better compressive strength than mixes produced with AL/FA=0.4.
- Comparatively higher compressive strength can be obtained for the mortar mixes cured at 80°C for 24 hours than that of the mixes cured at ambient temperature and water. When heat is applied to the samples for specified time, the reaction rate between fly ash and AL will be faster and strength providing geopolymeric structures formation will be also proper. Therefore, heat cured samples exhibited higher strength.
- From the micro-structural studies using SEM images, it can be concluded that lower AL/FA ratio may not be sufficient in the mixes to get reacted for forming geopolymeric products because lot of un-reacted fly ash particles are observed for the samples taken from the mixes produced with the consideration of AL/FA = 0.4.
- Dense geopolymeric matrix had been formed when AL/FA = 0.6 was considered to produce the fly ash geopolymer mortar mixes. As a result, higher strength is achieved for those mixes.
- Improvement of the properties of fly ash based geopolymer paste was achieved by incorporating GGBFS in the mixes. Significant reduction in setting time and increase in compressive strength had been obtained by adding GGBFS at different percentage level. Therefore, drawbacks of fly ash based geopolymer paste related to setting time can be eliminated with the usage of GGBFS.

- Higher compressive strength of fly ash based geopolymer paste incorporated with GGBFS was achieved due to the dense formation of the silico-aluminate structures from the reaction between AL, FA, and GGBFS.

6.6 CONCLUSIONS ON DRYING SHRINKAGE OF GEOPOLYMER MORTAR MIXES

- Higher RFA content in mixes, higher ratio of SS solution to SH solution in AL and higher AL/FA ratio resulted in more change in length and higher drying shrinkage value. But, lesser drying shrinkage value was observed for those samples of mortar mixes produced with higher concentration of SH solution in AL.
- Less remarkable change in length of the cast samples in longer dimension was observed after 28 days of air curing and for the heat cured samples, this duration was 3 days.
- For air cured samples, highest drying shrinkage value of 2543 micro-strain has been observed for the samples of the mortar mixes produced with 50% RFA, the ratio of Al to FA as 0.6 and AL consisted of 6M concentration of SH solution with the SS/SH ratio as 2.5.
- For heat cured samples, highest drying shrinkage value of 2288 micro-strain has been observed for the samples of the mortar mixes produced with 50% RFA, the ratio of AL to FA as 0.6 and AL consisted of 6M concentration of SH solution with the SS/SH ratio as 2.5.

6.7 CONCLUSIONS ON ANN PREDICTION MODEL

Based on the experimental values, models are generated to predict the properties of the fly ash based geopolymer mortar mixes. From the performance evaluation by different statistical indices, it can be concluded that most of the models perform with a greater level of precision. ANN model 4 - 9 - 1 using tansig as transfer function is found to be the best model to predict water absorption capacity of different fly ash based geopolymer mortar mixes. To predict the compressive strength of different fly ash based geopolymer mortar mixes at different ages, ANN model 5 - 12 - 4 using logsig as transfer function is found to be the best model. The ANN prediction model (5 - 12 - 1) using tansig as transfer function performs better for forecasting drying shrinkage value of different fly ash based geopolymer mortar mixes.

6.8 LIMITATIONS OF THE STUDY

- Quality of RFA may not be similar always as the properties of the parent concrete specimens are unknown. But the gradation of RFA and combination of RFA and NFA has been tried to maintain in such a way that it conforms zone II of fine aggregate as of IS: 383-2016.
- Solubility of SH pellets in water depends on temperature. Therefore, AL may act differently with the variation of temperature. Evengeopolymeric reaction also is influenced by temperature too. Therefore, variationin temperature has been ignored in this research work.

6.9 RECOMMENDATIONS

From the observations of this experimental work, it can be recommended to use concrete wastes as an alternative material for the natural fine aggregate in the construction industry. RFA can be used upto a certain percentage level to produce mortar/concrete mixes with desired properties. Fly ash based geopolymer mortar mixes can be produced with RFA partially as fine aggregate to use different purposes. This can be used to form the prefabricated blocks, paver blocks, bricks etc. Using of RFA effectively with geopolymer, which is an eco-friendly alternative binder material, helps to protect the natural resources and save the environment efficiently.

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