DEVELOPMENT OF FIBRE REINFORCED GEOPOLYMER CONCRETE (FRGC) CURED UNDER AMBIENT TEMPERATURE FOR STRENGTHENING AND REPAIR OF EXISTING STRUCTURES

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"To the memory of my father, Kadhim Haloob, It is your shining example that I try to emulate in all that I do. To my mother, brothers and sisters for their everlasting concerns, To my wife, without her supports it couldn't have been possible to make such a thing, And to my little son, for his patience and his motive beautiful smiles"

ABSTRACT

Most of the previous research on plain and fibre reinforced geopolymer concrete (FRGC) has concerned on the properties of geopolymer mixtures hardened under heat curing conditions, which is a severe limitation for on-site, cast-in-place applications. This study focuses on the material and structural properties of novel fibre reinforced geopolymer concretes cured under ambient temperature. The overall aim of the study was to develop and test a more environmentally sustainable concrete material with improved structural characteristics, which utilises waste rather than primary mineral products, suitable for cast-in-place applications and for the structural strengthening of existing buildings.

In the first part of this thesis, the material behaviour of FRGC cured under ambient temperature was examined. Initially, the work identified the role of various parameters which may affect material compressive strength, in order to enhance overall performance. In addition, the mechanical and microstructural properties of geopolymer mortar with different slag contents and variant silica fume types (densified, undensified and slurry) were assessed. Following this, the effect of slag content and silica fume particle size on the properties of steel fibre reinforced geopolymer composites (SFRGC) was examined. The optimum FRGC mixtures were further investigated in term of its durability characteristics and mechanical properties, in order to provide strain hardening characteristics. In the examined mixes, different fibre types, aspect ratios, and volume fractions, and its comparison with Portland cement based conventional concrete, have been assessed and appropriate mixtures have been identified with multiple fine cracks and strain hardening in tension.

In the final part of the thesis, the structural behaviour of FRGC is examined at larger scale application. PVA and steel fibre reinforced geopolymer concrete mixtures were used as strengthening and repair materials for the protection of steel bars in a new material layer, and for subsequent improvement of the flexural strength of existing beams. Large scale beams strengthened with additional FRGC layers reinforced with steel bars have been examined. Also, an additional investigation was conducted in beams where part of the concrete cover at various depths was replaced by FRGC. In all the examined cases respective beams with conventional concrete were examined in order to evaluate the efficiency of the proposed technique. Accelerated corrosion tests were performed using the induced current technique by applying a nominal 300 mA constant anodic current. The results of this investigation showed significant improvements in the structural performance of the examined beams following strengthening or repair with FRGC. The outcomes of the experimental work indicate that FRGC considerably enhanced both the flexural strength capacity and the durability of strengthened and repaired reinforced concrete elements.

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LIST OF ABBREVIATIONS

AAF	Alkali activation fly ash
AAS	Alkali activation slag
ACI	American Concrete Institute
ASTM	American Society of Testing Material
AS	Australian Standard
BS EN	British Standard European Norm
C-A-S-H	Calcium aluminium silicate hydrate
CaO	Calcium Oxide
C-S-H	Calcium silicate hydrate
DSC	Differential scanning calorimetry
DSF	Densified silica fume
ECC	Engineering cementitious composite
EDS	Energy dispersive spectroscopy
FA	Fly Ash
FRC	Fibre reinforced concrete
FRGC	Fibre reinforced geopolymer concrete
FTIR	Fourier transform infrared
GGBS	Ground Granulated Blast-Furnace Slag
GFRGC	Glass fibre reinforced geopolymer concrete
K-silicate	Potassium silicate solution
КОН	Potassium hydroxide
MS	Modulus of activator
MR	Molar ratio
N-A-S-H	Sodium aluminium silicate hydrate
NS	Nano silica
OPCM	Ordinary Portland cement mortar
PET	polyethylene terephthalate fibre
PVAFRGC	polyvinyl alcohol fibre reinforced geopolymer concrete
SEM	Scanning electron microscope
SF	Silica Fume
SFRGC	Steel fibre reinforced geopolymer concrete
SH	Sodium hydroxide (NaOH)
SHCC	Strain hardening cementitious composite
SHGC	Strain hardening geopolymer concrete
SS	Sodium silicate (Na ₂ SiO ₃)
SSF	Slurry silica fume
ST	Steel fibre
USF	Undensified silica fume
V_{f}	Volume fraction
w/c	Water to cement ratio
R	Coefficient of correlation
\mathbb{R}^2	Coefficient of determination

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DECLARATION

I declare that the research contained in this thesis, unless otherwise formally indicated within the text, is the original work of the author. The thesis has not been previously submitted to this or any other university for a degree, and does not incorporate any material already submitted for a degree.

Candidate: Mohammed Kadhim Haloob Al-Majidi

Signature: Lees

Dated: 20 / 09 /2017

CHAPTER 1:

INTRODUCTION

1.1 Background:

Concrete is a composite material made of cement, water and mineral aggregate. Ordinary Portland Cement (OPC) has traditionally been used as the primary binder material to produce concrete. The use of conventional concrete is greater than any other man-made material on the planet and concrete is the second-most utilised substance after water (Duxson and Provis, 2008). As the demand for infrastructure development increases, the consumption of concrete as a major construction material also increases. The quantity of OPC produced has increased year upon year, the global production of cement amounted to 4.6 billion metric tonnes in 2015 (Figure 1.1) (Association, 2016). Figure 1.1 illustrates cement production by region and country, highlighting that developing countries such as China, India and many African nations are growing their cement production compared to those in Europe (The European Cement Association, 2014).

It is well-known that the production of OPC consumes considerable amounts of virgin materials, resources and energy and causes substantial quantities of carbon dioxide to be emitted to the atmosphere. Global cement production accounts for 5-7% of worldwide industrial energy consumption (Turner and Collins, 2013). The emissions of greenhouse gases through cement production range from 0.66 to 0.82 kg of CO₂ emitted per kilogram cement manufactured, or about 2.8 billion tons of greenhouse gas emissions annually (Vora and Dave, 2013). These emissions are most commonly related to calcination of the raw material (limestone) and high energy consumption through manufacturing (Gartner, 2004). In addition to CO₂, the cement industry emits SO₂ and NOx, which can generate additional climatic and air pollution effects, and acid rain.

Climate change is a global issue, and many countries are working to reduce their emissions of greenhouse gases to move towards the concept of sustainable development. For example, the UK government targets an 80% reduction in greenhouse gas emissions by 2050. Many governments around the world are beginning to tax CO₂ emissions and promote more sustainable and environmentally friendly materials (Rickard, 2012b). Although the use of OPC seems unavoidable for the foreseeable future, much research is ongoing to find an alternative, more environmentally-friendly, materials to fully or partially replace OPC in concrete production (Lee and Lee, 2013). This is the main driver for the development of geopolymer concretes and geopolymer cement of all types, with their much lower carbon dioxide footprint (Davidovits, 2011).



Figure 1.1: World Cement Production 2015, by region and main countries, % 4.6 billion tonnes total (CEMBUREAU)(Association, 2016).

Geopolymer concrete is a relatively new material based on industrial by-products such as fly ash, slag, Metakaolin, rice husk ash and silica fume. Geopolymer materials have the potential to play an important role in the context of sustainability and environmental issues. Their use could reduce the carbon dioxide emissions associated with the manufacturing of cement by 80% (Islam *et al.*, 2014; Al-Majidi *et al.*, 2016b). In addition, one of the main concepts of sustainable construction could be achieved through reducing the overuse of virgin material such as limestone. Millions of tons of industrial waste are generated every year and its utilization is an attractive alternative to disposal to overcome storage problems, disposal costs and potential pollution issues related to storage and environmental disposal. All of these issues are reduced or even eliminated with use of geopolymer materials, along with the achievement of resource conservation (Islam *et al.*, 2014).

The term geopolymer was firstly used by the French researcher Davidovits in 1978, who researched the use of kaolinite (Al₂Si₂O₅(OH)) and alkaline activators. Geopolymers are inorganic materials rich in silicon (Si) and aluminium (Al) that react with alkaline activators to become cementitious material (Ryu *et al.*, 2013). The most widely adopted alkaline activators are a hydroxyl, usually sodium hydroxide (NaOH) or potassium hydroxide (KOH), and a glassy silicate, consisting of sodium silicate or potassium silicate, which are used individually or in combination. The alkali activation of aluminosilicate materials is a chemical process that transforms partially or wholly vitreous structures into compact cementitious skeletons. There are many factors which effect the nature and rate of chemical reactions in, and the rate of strength development of, geopolymer concrete such as mixing steps, mixing time, curing conditions, alkaline type, alkaline concentration and the source of raw materials (Joseph and Mathew, 2012; Lee and Lee, 2013; Ryu *et al.*, 2013; Turner and Collins, 2013; Al-Majidi *et al.*, 2016b).

It has been reported that geopolymer concrete cured at high temperatures can obtain comparable performance (in terms of mechanical, physical and durability properties) to conventional concrete, such as high compressive strength, low porosity, fire resistance, rapid setting and hardening, and low shrinkage (Chi, 2012; Lee and Lee, 2013; Deb et al., 2014). Fly Ash and Ground Granulated Blast-Furnace-Slag (GGBS) are the most commonly used solid waste materials in geopolymer concrete (Al-Majidi et al., 2016b). Fly ash is a by-product from thermal power stations and GGBS is formed during the process of pig iron manufacture from iron ore. Both of these products are aluminosilicate materials rich in Si and Al but their physical properties and reaction product are quite different. The main reaction product of alkali-activated cement for slag is a C–S–H gel while for class F fly ash it is the amorphous hydrated alkali-aluminosilicate (Chi and Huang, 2013; Ismail et al., 2013). Another example of a by-product material used as a cementitious material is silica fume, which is produced during the manufacture of silicon metal and ferro-silicon alloys. It is rich in silica with variable particle size (depending on its physical form: densified, undensified, slurry and nano silica), and, having a greater surface area and higher SiO₂ content, has been found to be more reactive than fly ash (Chi and Huang, 2013; Jang *et al.*, 2014). It has become vital to use silica fume to produce high strength geopolymer concrete as it improves the bond strength between the cement paste and the aggregate. It also increases the mechanical strength of concrete because of its pozzolanic activity (Köksal *et al.*, 2008).

Fly ash based geopolymer concrete cured under ambient temperatures however has slow pozzolanic reaction rates, causing low early strength development. This might be because of the unavailability of sufficient quantities of Ca(OH)₂ to react with fly ash particles (Barbhuiya *et al.*, 2009; Görhan and Kürklü, 2014; Jang *et al.*, 2014) or because the free silica in fly ash is trapped in the interior part of its particles. Therefore, it is vital to improve the early strength of ambient-temperature cured fly ash concretes to meet the technical serviceability requirements relating to both their engineering and durability properties. It is possible to improve the early strength of geopolymer concrete by combining materials, in binary and ternary blends, which accelerate the pozzolanic reactions. No studies have been published (to the author knowledge) the use of binary and ternary blended geopolymer concrete (in contrast with the case of normal concrete). Hence, the benefits of blending for fly ash based geopolymer concrete, particularly those cured at ambient temperatures, remain unclear.

Separately, concrete shows brittle behaviour due to its low tensile strength, which is only about 10% of its compressive strength, and so concrete cracks when it is subjected to tensile stresses. Many researchers have found that fibre addition is an effective method to improve mechanical performance under tensile stresses and control shrinkage by bridging cracks, and changing the brittle behaviour of concrete to ductile or quasi-ductile, with significant improvement in tensile strength, tensile strain, toughness and energy absorption capacities (Bernal *et al.*, 2010; Shaikh, 2013a). Hence, fibre reinforced concrete (FRC) has been widely used in areas such as industrial pavement, shotcrete for early stage tunnel linings and as a partial replacement for conventional reinforcement (rebars or welded mesh) because of the possibility of stress redistribution and strengthening of the existing structures (Slater *et al.*, 2012; Soutsos *et al.*, 2012). Recently, there have been efforts to replace the cement based binder in current FRC with geopolymer binder (Alomayri and Low,

2013; Nematollahi *et al.*, 2014). However, the evolution of fibre reinforced geopolymer concrete (FRGC) is relatively new in the field of construction materials.

Durability is a vital parameter of concrete as due to its fundamental relationship with the serviceability life of the concrete structure. The reinforced concrete building must be able to resist the mechanical actions, physical and chemical aggressions they are subjected to during their expected service life (Ganesan *et al.*, 2015). The previous researches have shown that geopolymer concrete cured under elevated temperature has superior durability in term of chemical resistance, thermal resistance and chloride ion penetration (Bakharev, 2005c; Sathia *et al.*, 2008; Rajamane *et al.*, 2012; Ganesan *et al.*, 2015). No study seems to have done so far (to the author's knowledge) to evaluate the durability of geopolymer material cured under room temperature. Moreover, the influence of incorporating randomly distributed fibres on the durability characteristics of geopolymer cured under room temperature was not studied so far. Considering this gap in the literature, an attempt has been made in the research to investigate the durability characteristics of plain geopolymer and fibre reinforced geopolymer composite cured under room temperature and its comparison with the conventional concrete.

Repair and strengthening of reinforced concrete (RC) members are becoming a major part of construction activities to extend their service life (Martinola *et al.*, 2010; Mourad and Shannag, 2012; Iqbal *et al.*, 2016). Repair refers to modification of structure, damaged in its appearance or serviceability, to restore, partly or wholly, the pre-existing characteristics of serviceability, load- bearing capacity and if necessary, to improve its durability. It is generally include removal of damaged concrete and replacement of new concrete. While, structural strengthening may be required for increasing load capacity of structures, seismic resistance, and supporting additional live or dead loads not included in original design (Ruano *et al.*, 2014; Lampropoulos *et al.*, 2016). There are a huge number of structures around the world that have reached a critical age with increasing signs of deterioration and minimized functionality (Mourad and Shannag, 2012). Moreover, there are important infrastructural elements, such as bridges or tunnels, which have to be repaired to avoid the social costs related to their demolition and the reconstruction of new structures. Accordingly, there is a large demand for repair work to enhance the quality of concrete structures for the duration of their service lives and reduce cost (Koteš *et al.*, 2013). The effects and benefits of strengthening and repair of existing reinforced concrete structure by using fibre-reinforced geopolymer concrete remain relatively unexplored to date.

1.2 Aims and Objectives

The excellent environmental and mechanical properties of geopolymer concrete have encouraged its use as a construction material. Previous research has focused on investigating geopolymer concrete cured under elevated temperature, which runs somewhat counter to its use as a low CO₂ material, as this consumes significantly more energy compared to room-temperature curing. The long heat curing period also limits the industrial field application of the geopolymer. The realization of room temperature curing of geopolymer concrete is critical for various commercial uses. The principle aim of this research is to address the knowledge gaps outlined above, by developing fibre reinforced geopolymer concrete cured under ambient temperature that can be used in practice for strengthening and repair of existing structures.

Thesis Outline and Objectives;

The research was organised in four phases, proceeding from material to structural elements:

- Material stage; focusing on the development of strength, durability and microstructure in ternary blend geopolymer materials, produced by mixing fly ash, slag and silica fume. The effect of fibre addition was also assessed, with research examining the durability and strength of varying lengths, volumes and types of fibre (steel, PVA and glass) in fibre reinforced geopolymer concrete (FRGC).
- Structural stage; Using fibre reinforced geopolymer concrete for retrofitting structural application as a repair / strengthening material under corrosive conditions.

The detailed objectives of this study are as follows:

1- To develop a user friendly geopolymer mortar cured under room temperature by identifying a suitable mix design. The development of user friendly (noncorrosive) will promote easier (safer) handling treatment, cured under ambient temperature and cast-in-place applications.

- 2- To enhance the strain hardening characteristics of fibre reinforced geopolymer concrete to make it appropriate for structural interventions.
- 3- To evaluate the durability of the examined mixtures in order to identify the suitability of FRGC material for repair and structural applications.
- 4- To evaluate the efficiency of FRGC as repair material in terms of flexural load carrying capacity and the durability of RC beams where a part of the concrete cover at various depths has been replaced by FRGC.
- 5- To assess the efficiency of strengthening technique of RC beams by using FRGC additional layer in terms of flexural load capacity and durability performance.

The above objectives outline the scope of the thesis. The use of geopolymer concrete significantly reduces the carbon dioxide emissions associated with cement production. This is an additional benefits of the proposed application but this environmental aspect has not been investigated in this thesis.

1.3 Thesis Structure

This thesis is organized into nine chapters and appendices (Figure 1.2). The initial chapter provides an overview of the work, current knowledge gaps, and describes the motivation for developing cement free geopolymer as an alternative binder to ordinary Portland cement (OPC). The chapter also presents the research aims and objectives. Subsequent chapters cover in detail specific elements of the research:

Chapter 2 reviews the history and the environmental impact of ordinary Portland cement industries and reviews the existing knowledge of geopolymers, the geopolymerization mechanism, and the properties of geopolymer mortar/ concrete. The chapter describes the types of by-product materials which are suitable for application as a geopolymer binder, their uses and their availability. Literature on the behaviour of fibre reinforced cementitious composites (FRCC), and repair and strengthening techniques for reinforced concrete beams using FRCC are also presented.

Chapter 3 reports the overall research methodology and experimental details including materials used, sample preparation and testing programs to assess the mechanical and structural properties of plain geopolymer, fibre reinforced geopolymer

composite, and their potential applications. Subsequent chapters then review the results from these tests.

Chapter 4 examines the fresh and hardened properties of binary blended geopolymer mixtures, looking at the setting time, flow table results, compressive strength, and tensile strength. This chapter also evaluates the physical and micro-structure of the examined mixtures by scanning electronic microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and thermal analysis (DSC).

Chapter 5 examines the fresh, microstructural and strength development properties of ternary blended (fly ash, slag and silica fume) geopolymer mortar and the effect of the geopolymer matrix composition on the microstructure and mechanical performance of steel fibre reinforced geopolymer concrete (SFRGC).

Chapter 6 examines the strain hardening characteristics of geopolymer matrices reinforced by varying fibre types, aspect ratio and volume fractions, using compressive strength, tensile strength and flexural strength tests.

Chapter 7 evaluates the durability properties of fibre reinforced geopolymer concrete. The chapter discusses the shrinkage characteristics, corrosion resistance, chloride penetration depth and chemical resistance of the fibre reinforced geopolymer material.

Chapter 8 assesses the structural performance of FRGC applied as a composite repair material, both in a composite beam and as a repair/jacketing material. The effect of corrosion on the flexural response of the repaired /strengthened RC beam are also discussed.

Chapter 9 summarises the overall conclusions from this study and also make recommendations for future research in this area.

References list of References has been listed.

Appendices List of the published work has been listed at the end of the thesis.



Figure 1.2: Outline of the thesis.
CHAPTER 2:

LITERATURE REVIEW

2.1 Background of concrete technology.

Concrete is a material used in building construction, consisting of a hard, chemically inactive particulate material, known as an aggregate (sand and gravel), that is bonded together by cement and water. The cement is a mixture of diverse minerals which when mixed with water, set and rapidly become hard, binding concrete components together into a solid mass.

The first leading concrete users were the Egyptians around 2,500 BC, when they used lime and gypsum cement. The Chinese built the Great Wall between 200-300 BC by using cementitious materials to bond bamboo together, while the Assyrians and Babylonians used clay as a bonding substance or cement and the Greek civilisation used lime mortars in about 800 B.C. (Steiger, 1995). Joseph Davidovits notes that the pyramids and temples of the Old Kingdom in Egypt were constructed using reagglomerated limestone, with lime and zeolite forming materials, which is termed geopolymer cement, rather than quarried and hoisted blocks of natural limestone. However, many researchers believe that truly cementitious materials were first developed by the Romans in about the third century B.C. They used Volcanic ash (Pozzolana) mixed and reacted with lime mortar, sand, gravel and water to produce a consolidated hard substance similar to recent concrete. Some of the most famous masterpieces built by the Romans using the ancient concrete are the Coliseum and the Pantheon (Shaeffer, 1992; David Moore, 2003).

In the 1700s John Smeaton, an English Engineer, generated a significant advance in concrete technology through the production of hydraulic lime, a lime that hardens under water. He used this material to rebuild the Eddystone Lighthouse in Cornwall, England. The 19th century saw rapid advances in concrete technology all over the world, notably in 1824 the English inventor Joseph Aspdin discovered Portland cement, which has remained the main cement used in concrete production. Aspdin produced Portland cement by burning a mixture of ground limestone and clay into clinker, then pulverizing it into powdered cement. The burning process changed the

chemical characteristics of the material and produced a better cement quality than plain crushed limestone (Bye, 1999).

Another major advance occurred in 1849, from the French gardener Joseph Monier. Monier reinforced garden pots and tubs with iron mesh and developed the concept of reinforced concrete. Monier received a patent for his invention at the Paris exhibition in 1867. Reinforced concrete combines the tensile strength of steel with the compressive strength of concrete. Monier promoted reinforced concrete for use in the building of many commercial structures such as railway ties, pipes, floors, arches and bridges. The late 19th and early 20th centuries saw construction of the first reinforced concrete bridge (Alvord Lake Bridge in the USA) in 1889, and the oldest American concrete street, placed in 1891 by George Bartholomew in Bellefontaine, Ohio. In 1903 the world's first high rise concrete building was constructed in Cincinnati, Ohio. Other important concrete structures constructed in the later part of the 20th century include the Grand Coulee Dam in Washington, completed in 1942, which is the largest concrete structure ever built (Armstrong, 2001; Gromicko and Shepard, 2006). In the years following, the tallest reinforced concrete building in the world, built in Chicago, Illinois, in 1992 and the world's tallest structure (as of 2017), the Burj Khalifa in Dubai, was built using reinforced concrete.

Separately from Ordinary Portland Cement, the first work in the area of alkaliactivated slag was carried out by Feret, (1939) and Purdon, (1940). It was not until 1959 however that Glukhovsky (1959) developed an alkaline aluminosilicate cementitious system, termed "soil silicates". In 1972, Joseph Davidovits discovered three-dimensional silico-aluminate materials which synthesised at low temperature over a short duration using various naturally occurring minerals. He termed these materials geopolymers (mineral polymers resulting from geochemistry) (Davidovits, 1988). Richard Heitzmann and James Sawyer in 1984 developed early high strength geopolymer concrete at Lone Star Industries Inc in the United States, and termed this Pyrament Blended cement (PBC). The material is prepared by mixing 80% blended ordinary cement with 20% of geopolymer raw material, which generates a product with high acidic resistance and short setting time, which is used in many construction industry applications (US Army Corps of Engineers, 1985). PBC is an effective material for repairing industrial pavement, and runways made of concrete, because 46 hrs hardening is sufficient to reach strengths of 20MPa (high enough to accommodate a landing aeroplane) compared with ordinary concrete which needs several days to reach this strength. In 1994 a US Army Corp Engineers study show that PBS had better quality than was expected and it is listed as being used in 57 military installations in the USA and other countries (Davidovits, 2011). Recently, there have been a number of studies on geopolymer concrete in order to advance understanding of geopolymers and to overcome the obstacles to using geopolymer concrete on an industrial scale (see below).

2.2 Main issues concerning Ordinary Portland cement (OPC) concrete.

2.2.1 Environmental issues.

Concrete is a versatile material which is used as a construction material in many applications because it can be poured and take any shape, from nuclear radiation shields to playground structures and from bridges to yachts (Foley, 2005). Ordinary Portland cement is a fine powder commonly used as a binding material to bind fine sand and coarse aggregates together in concrete. Ordinary Portland Cement is manufactured in three stages; raw-meal preparation, production of cement clinkers by heating the raw material at 1400 °C to 1600 °C, and cement milling into fine powder (Foley, 2005). Figure 2.1 shows an evaluation of cement production from 2001 to 2015 by region. Notably, production in Asia and Africa has risen steadily through this timeframe to indicate an increase of 175% and 155%, respectively, on 2001 data. The increasing demand for concrete will lead to a reduction in the availability of sources such as limestone, chalk, and shale or clay, and consume enormous amount of energy. Struble and Godfrey, (2004) estimated that 4.88 MJ/kg of energy is consumed in the production of OPC with 85% of this energy used in heat treatment of the raw material to generate cement clinker in the klin. The residual 15% is used for the preparation of raw materials entering the kilns, and grinding the cement clinker that is fired from the kiln (Ng, 2011).



Figure 2.1: Evaluation of World cement production by region from 2001 to 2015 (Association, 2016).

Further, OPC production causes large amounts of CO_2 release from the calcination reaction through the manufacturing process (see also chapter one). According to Rodríguez *et al.*, (2008) the calcination of CaCO₃ to make 1 ton of Portland cement emits 0.53 tons of CO₂ into the atmosphere, and if the energy used in the production of Portland cement is carbon fuel then an additional 0.45 tons of CO₂ is produced. Therefore the production of 1 ton of Portland cement generates approximately 1 ton of CO₂ (Adam, 2009). Therefore, the production of cement poses serious environmental issues and makes a significant contribution to global climate change.

2.2.2 Durability Issues.

In designing concrete structures, durability is considered as one of the most important properties beside the ability of the structures to resist all relevant loads (Abdelkader *et al.*, 2010). Insufficient durability of conventional construction materials means that structures deteriorate much earlier than their design service life, particularly in marine or industrial environments (Malhotra and Mehta, 2005; Singh *et al.*, 2015b). Also, using inappropriate construction materials lead to high conservation cost to maintain the concrete structure and, in some cases, the maintenance costs over the initial construction costs, troublesome, or even impossible to apply (Zhang *et al.*, 2010a).

OPC concrete structures exposed to aggressive environments can be durable materials with proper design and production. However, conventional concrete can suffer from chemical deterioration due to attack from aggressive agents such as chloride, sulphate, and acid (Seleem et al., 2010). This is due to chemical reactions between aggressive solutes and cement hydration products and the reinforced steel bars in concrete, although reactions can also happen with the aggregate, i.e. alkali aggregate reaction (Adam, 2009). The mechanisms are mainly due to the carbonation of cement hydration products $Ca(OH)_2$ in wet environments in the presence of Cl^- , Mg^{2+} and $SO4^{2-}$ ions. The carbonation product CaCO₃ can be further dissolved in contact with water. Consequently, capillary pores allow more corrosive ions to move in, and meanwhile the decreased alkalinity of the cement matrix due to the loss of Ca(OH)₂ further increases the potential for chloride corrosion. Chloride is one of the most problematic ions for reinforced concrete (Zhang et al., 2012). Besides this, other factors can impact on concrete durability and strength, particularly in marine structures, such as crystallization pressure of salts within concrete (if one face of the structure is subject to wetting and others to drying conditions), frost action in cold climates, corrosion of embedded steel in reinforced or prestressed members, and physical erosion due to wave action and floating objects (Seleem et al., 2010). In order to extend service life, the concrete should have low permeability and high resistance to chloride ingress (Cheewaket et al., 2010; Zhang et al., 2010b).

2.3 By-Product materials.

2.3.1 Fly Ash.

The term fly ash is used for fine particulate solid materials precipitated from the boiler of industrial furnaces burning solid fuels, mainly from coal fired thermal power plants (ASTM C618-08, 2008). Fly ash is produced by burning ground coal in furnaces at high temperature (around 1500 °C), where the coal in suspension is burnt instantaneously. Exposure of lighter incinerated components to rapid cooling as they are carried away from the burning zone in the boiler by flue gases leads to ash solidification into fine spherical particles. Finally, these fly ash particles are collected from the flue gas by means of electrostatic precipitators (Davidovits, 2011). This is the material termed pulverized fuel ash - the heavier unburned material which drops to the bottom of the furnace is termed bottom ash which is not suitable for use as cementitious material.

In term of availability of fly ash, coal is the largest fuel source and still plays a vital role in generation of electricity, around 41% of global electricity. Coal is the second source of primary energy in the world after oil, and the primary source of electricity generation. Since the beginning of the 21st century, coal has been the fastest-growing global energy source. There has been a recent 60% growth worldwide coal consumption from 4,762 million tonnes in 2000 up to 7,697 million tonnes in 2012 (International energy Agency, 2015). According to American Coal Ash Association Educational Foundation, (2015), the Unites States in 2007 produced 131 million tons of coal combustion products, but only 43% have been beneficially re-used; around 75 million tons were disposed of into landfills. In Australia only 32% of fly ash was beneficially re-used. As the requirements for electricity increase, a huge amount of fly ash is generated and disposed of in landfills or storage lagoons. It is thus anticipated that fly ash will be available well into the future (Heidrich, 2003). Table 2.1 shows the coal consumption percentages in some countries. A similar trade group in Europe estimates that only around half of the 44 million tons of fly ash produced in the "EU 15" in 2006 was reused (Association., 2010); in India, more than 130 million tons are currently produced, with around half recycled (Chatterjee, 2010); while in China 150 million tons are produced, of which two-thirds was recycled (Cao, 2008). The United States, Europe, India, and China are the four biggest producers of coal worldwide; their consumption of fly ash varies widely by region, with some areas recycling no fly ash, and other areas using more than is produced every year, taking from stockpiles of other regions (Sakulich, 2011).

Coal in Electricity Generation				
South Africa 93% Poland 87% RP China 79%				
Australia 78% Kazakhstan 75% India 68%				
Israel 58% Czech Rep 51% Morocco 51%				
Greece 54%	USA 45%	Germany 41%		

Table 2.1: Coal in Electricity Generation (IEA, 2012).

2.3.1.1 Characteristic of Fly Ash.

Fly Ash is an aluminosilicate material, in which the main chemical constituents are Silica (SiO₂), Aluminium Oxide (Al₂O₃), Calcium Oxide (CaO) and Iron Oxide (Fe₂O₃) (Somna *et al.*, 2011). Fly ash can take various forms and the exact composition of fly ash varies from one plant to another and from day to day in the same plant. This

is since the chemical and physical characteristics of fly ash particles are a function of the type of coal (anthracite, bituminous or hard coal, lignite, and brown coal), the combustion conditions, and post-combustion cooling. During the combustion process, the heat causes inorganic minerals present in coal to become fluid or volatile or to react with oxygen. During cooling, these may form crystalline solids, spherical amorphous particles or condense as coatings on particles (Kutchko and Kim, 2006).

According to the ASTM C618-08, (2008) standard, there are two classes of fly ash based on Calcium Oxide (CaO) content, as presented in the Table 2.2. The only limit placed on the composition of the fly ash by the ASTM C618-08, (2008) and BS EN 450-1, (2012) specifications is a maximum allowable loss-on-ignition (LOI). LOI indicates the amount of unburned carbon present in the fly ash. The LOI limit in ASTM C618-08, (2008) is 6% for both class F and C fly ash, while the BS EN 450-1, (2012) classifies fly ash into three categories A, B and C based on LOI content. LOI weight percentage in Fly Ash under Category A has to be less than 5%, category B between 2% to 5% and Category C from 4.0 to 9.0% (which is not permitted in the UK concrete as the LOI upper limit under BS 8500 is 7.0% (Thomas, 2007). Fly Ash consists of mainly spherical particles with particle sizes between 1 and 150 microns, which is similar to, or slightly less than, that of Portland cement. The finest fraction improves mix grading and two categories of fly ash are permitted under BS EN 450; Category N and S, for which not more than 40% and 12% should be retained on a 45 micron sieve respectively (Lafarge Tarmac, 2014).

Category	Description	Chemical	
		Requirements	
Low Calcium fly Ash Class F	CaO <10%, Fly ash normally produced from burning anthracite or bituminous coal that meets the applicable requirements	$\begin{array}{l} SiO_2 + Al_2O_3 + Fe_2O_3 \\ \geq 70\% \end{array}$	
High calcium fly ash Class C	CaO >10%, Fly ash normally produced from lignite or sub bituminous coal that meets the applicable requirements	$\begin{array}{l} SiO_2 + Al_2O_3 + Fe_2O_3 \\ \geq 50\% \end{array}$	

Table 2.2: Types of Fly Ash (ASTM C618-08, 2008).

Element	Typical range of values for fly ash
SiO ₂	38-52
Al ₂ O ₃	20-40
CaO	6- 16
MgO	1-3.5
Na ₂ O	0.8- 1.8
K ₂ O	2.4-4.5
TiO ₂	0.9-1.1
SO ₃	0.35-2.5
LOI	3-20
pH	9-12

Table 2.3: Typical range of analyses from UK fly ash.

2.3.1.2 Utilization of Fly Ash in Concrete.

Fly Ash is very fine-particulate material with grey colour, depending on it is source. For many years it has been usual practice to replace Ordinary Portland cement by fly ash in the range of 10 to 30% by mass of the total cementitious material (Crouch, 2007; Zhou *et al.*, 2012). The behaviour of the fresh state of the concrete and the mechanical properties and durability of hardened concrete are significantly affected by the incorporation of the fly ash into the mixture. The physical properties of fly ash give several benefits when it is used in concrete. It is well known that fly ash improves the workability, rheology, and reduces bleeding of concrete as a result of a "ball bearing" action of the spherical and ultrafine fly ash particles (Lafarge Tarmac, 2014). Fly ash develops grading in the mixture by smoothing out the fine particle size distribution, and thus improves the particle packing of concrete as it fills up the gaps between larger particles and reduces the amount of manufacturing water required (Mehta and Monteiro, 2006). Chindaprasirt et al., (2005) examined the influence of varying finenesses of fly ash on pore structure and microstructure of fly ash cement pastes. These authors noted that the blended cement paste containing finer fly ash particles showed a denser matrix and resulted in higher compressive strength and lower total porosity.

Fly Ash also has pozzolanic activity due to it has high silica and aluminium content which reacts with calcium hydroxide during cement hydration, to produce additional Calcium Silicate Hydrate (C-S-H) and Calcium Aluminate Hydrate (C-A-H). These are effective in forming a denser matrix leading to higher strength and better durability (Supit *et al.*, 2014). It has also been noted that using fly ash concrete reduced the heat

of hydration, increased resistance to sulfate attack and reduced manufacturing cost. Fly Ash concrete was first used in mass concrete construction in the building of the Hungry Horse Dam, Montana, 1948. Fly ash concrete has been used in construction of a range of infrastructure including concrete roads, roller compacted concrete, dams, in the nuclear industry (e.g. use of high volume fly ash (HVFA) concrete in the Torness and Sizewell Nuclear Power Stations in the UK), in tunnels (e.g. the Channel Tunnel link between England and France), and in high rise buildings (e.g. the Petronas Tower of Kuala Lumpur, Sears Tower in United States, Prudential Building of Chicago).

2.3.2 Ground Granulated Blast Furnace Slag (GGBS).

Ground Granulated Blast furnace Slag (GGBS), or slag, is a molten non- metallic material that is produced from smelting of the siliceous gangue found in iron ore, the residue of coke combustion, the limestone and other added ingredients (Davidovits, 2011). Its production process by iron smelting at temperatures ranging from 1400°C to 1600°C. The slag floats over the pig iron at the bottom of the blast furnace, is rapidly cooled with water and then crushed and refined into fine cementitious powder.

Iron industries are spread widely across many countries in Europe, USA, Australia and Asia. In 2013, global steel production reached 1607 million tons and China recorded 48% of the total production (International Steel Statistics Bureau, 2015). Generally, 250-300 kg of slag are generated per tonne of iron, and 100-150 kg are produced per tonne of steel. Based on these estimates, UK and US production of slag since the mid-nineteenth century is 490–640 million tons and 2,100–2,700 million tons respectively. World production through this period is 12,200–15,900 million tons. Previously slag was considered as a waste material which was placed in large slag heaps in many areas, but nowadays slag is used as a component of concrete and road base. For example, the UK produces 2.2 million tons of slag annually and the vast majority of this is used as a cementitious addition supplied as separate material, conforming to EN 15167-1 'Ground granulated blast furnace slag for use in concrete, mortar and grout' (Jones, 2011).

GGBFS is an aluminosilicate material and the major chemical elements are silica (SiO_2) , aluminium oxide (Al_2O_3) and calcium oxide (CaO). The physical and / or chemical composition varies depending on the type of ore smelted, and the cooling

procedure of molten slag. For example, if the liquid slag is left to gradually air-cool, it is used as an aggregate, whereas blast furnace slag is used as cementitious material when it is rapidly quenched using large volumes of high-pressure fresh water sprays (civil and marine, 2007; Sakulich, 2011) . The typical chemical compositions of GGBS are shown in Table 2.4. In addition, slag has variable physical parameters, for instant; amount of glassy material, fineness and grain size distribution (Altan and Erdoğan, 2012).

Weight (%)	SiO ₂	AL ₂ O ₃	CaO	MgO	Fe ₂ O ₃
Slag	35	12	40	10	0.2

Table 2.4: Typical chemical composition of UK GGBS (Hanson UK).

GGBS is usually used in combination with normal Portland cement. The GGBS and cement are added into the concrete mixer as separate constituents. Where appropriate, the ratio of GGBS to cement can be varied according to the technical requirements for any particular application. GGBS can replace up to 70% or more of conventional cement and it is common practice in the UK for ready mixed concrete companies to produce concrete with a cementitious component of 50% GGBS and 50% Portland cement (Jones, 2011). It has been consistently reported that GGBS improves the compressive strength and durability properties of concrete, by reducing permeability, generating lower hydration, producing higher ultimate compressive strengths, increasing resistance to sulfate-acid attack and aggressive chemicals and producing better workability and finish ability than normal concrete (Altan and Erdoğan, 2012; Chi, 2012; Ismail *et al.*, 2013). GGBS concrete is used in many UK structures, such as the Queen Elizabeth II Bridge in Dartford, the Manchester Airport Second Runway, Norfolk and Norwich Hospital and more. Approximately 15 million tons of GGBS concrete are produced each year in the UK.

2.3.3 Silica Fume.

Micro silica is a by-product material of the smelting process in the silicon and ferrosilicon industries. The American concrete institute (ACI 116R) defines silica fume as "very fine non-crystalline silica produced in electric arc furnaces as a by-product of the production of elemental silicon or alloys containing silicon". In the early

1950s, Silica fume was used for the first time in concrete in Norway. Twenty years later, silica fume produced in large industrial scale by using filtering process. Silica fume has been identified as a very reactive supplementary pozzolanic and cementitious material because of its chemical and physical properties, and the addition of silica fume to concrete can significantly improve material strength and durability (Siddique, 2011; Ramezanianpour, 2014).

Figure 2.2 presents a schematic diagram of silica fume production by the reduction of high-purity quartz to silicon at elevated temperatures (up to 2000 °C) in an electric furnace. Coal, coke and wood chips were usually added to oxidizes and then condense in the low temperature zone to tiny particles consisting of non-crystalline silica. The fume that results from the furnace operation is collected in very large filters in the baghouse and then can be re-used in concrete instead of being landfilled (Bernd, 2006).



Figure 2.2: Schematic diagram of silica fume production (Siddique, 2011).

2.3.3.1 Characteristics and types of Microsilica.

The chemical composition of micro-silica is related strongly to the raw materials used and the furnace design, which controls the carbon content in the final product based on lower or higher ignition loss (Ramezanianpour, 2014). According to international standards (ASTM C 1240, 2005), the silica (SiO₂) content of silica fume for use in concrete must be at least 85%, with a small amount of magnesium, iron and alkali oxides present as shown in Table 2.5.

Oxides	Sandvik and Gjørv (2007)	Hooton and Titherington (2004)	Yazici (2008)
SiO ₂	92.1	96.65	92.26
AL ₂ O ₃	0.5	0.23	0.89
CaO	1.40	0.07	1.97
MgO	0.5	0.31	0.49
Na ₂ O	0.3	0.04	0.96
K ₂ O	0.7	0.56	1.31
Na ₂ O	0.3	0.15	0.42
SO ₃	-	0.17	0.33
LOI	2.8	2.27	-

Table 2.5: Chemical composition of silica fume samples (Siddique, 2011).

The colour of silica fume varies from dark to almost white powder depending on carbon content, somewhat similar to Portland cement or some fly ashes. Silica fume consists of very fine spherical particles, with more than 95% of the particles less than 1 μ m. Most particles are within a mean diameter of 0.2 μ m and have a very large surface area between 15000 and 30000 m²/kg (Bernd, 2006). Silica fume is available commercially in various forms based on the material handling, and these forms have advantages and disadvantages in terms of delivery, efficiency, production addition rate, and performance of concrete. The main commercial forms are as densified or undensified silica fume, or as water-based slurries (Siddique, 2011).

Undensified silica fume is referred also as product silica fume. It is an extremely fine powder with low loose density (200-300kg/m³ (Maas *et al.*, 2007)). It is available in bulk or in bags, based on the desire of the manufacturer to supply this form. It has not been used extensively in ready mixed concrete due to handling difficulties and higher transportation cost than other forms of silica fume. However, these issues could be somewhat overcome with properly designed loading, transport, storage, and batching systems (Ramezanianpour, 2014).

Densified Silica fume (compacted microsilica) is made by using undensified silica fume and some further treatment steps. The densification process significantly decreases the dust content compared to the as-produced silica fume. One procedure to manufacture densified silica fume is to place as-collected silica fume in a silo, and then compressed air is blown in from the lower end of the silo, causing the particles to tumble. As the particles tumble, they agglomerate. The heavier agglomerates fall to the bottom of the silo and are periodically removed. These products are dense enough (about 500 to 700 kg/m³) to be transported economically in bulk bags of 600 to 1200 kg, and in small bags of 22 to 55 lb (10 to 25 kg) (Diamond and Sahu, 2006).

Slurry silica fume is an aqueous suspension produced by blending silica fume powder with an equal quantity of liquid (water and water reducing agent). The suspension is easy to handle and pump which overcomes the difficulties related to transport and handling of undensified silica fume. Slurry silica fume has a specific gravity of 1400 kg/m³ which contains 700 kg/m³ dry material versus 130 to 430 kg/m³ for as-produced silica, and provides a good dispersion of silica fume particles in the concrete mix (Siddique, 2011).

Table 2.6: Physical properties of types of Silica Fume (Ramezanianpour, 2014).

Property	Particle	Bulk density [Kg/m3]			Specific	Surface
	Size	As	Slurry	Densified	gravity	area (BET)
		produced				$[m^2/kg]$
Value	< 1µm	130-430	1320-	480-720	2.22	13000-30000
			1440			

The incorporation of silica fume in concrete has many benefits such as improving the early compressive strength, generating higher tensile and flexural strength, and improving the concrete durability via its very low permeability to chloride and water intrusion, providing high resistance to chemical attack from chlorides, acids, nitrates and sulfates (Atiş *et al.*, 2005; Siddique, 2011; Oertel *et al.*, 2013). Many researchers have reported that the use of silica fume to produce high strength concrete (HSC) has become crucial because the physiochemical behaviour of silica fume develops the interfacial zone between the aggregate and cement (Chan and Chu, 2004; Ivorra *et al.*, 2010). The ultra-fine particles fill the voids between the composite particles and also silica fume has pozzolanic activity which converts the weak calcium hydroxide crystals into strong calcium silicate hydrate gel. With its use, the interfacial zone between tin durability (Atiş *et al.*, 2005; Köksal *et al.*, 2008; Toutanji *et al.*, 2010). In addition, silica fume is used in many other applications such as silica fume shortcrete, oil well

grouting, cementitious repair product, refectory castables and ceramics (Siddique, 2011; Siddique and Khan, 2011).

Ivorra et al., (2010) examined the addition of silica fume with an average SF particle size between 1 µm and 60 µm to carbon fibre reinforced concrete (CFRC). Their results indicate a different behaviour of concrete depending on the average SF particle size, with the highest compressive and flexural strength of CFRC achieved by addition of SF with average particle size between 5 and 15 µm. The addition of SF with particle size in the range of 5-75 nm did not lead to significant improvement of the strength proporties of CFRC. Oertel et al., (2013) focused on the influence of primary particle sizes and sizes of agglomerates of different amorphous silica forms (commercial silica fume and wet-chemically synthesized silica) in ultra-high-performance concrete (UHPC). These authors found that dispersion of agglomerated silica fume into primary particle sizes or the smallest agglomerates possible is mandatory to increase the reactive surface area for further improvement of the compressive strength. Haruehansapong et al., (2014) presented the effect of nano silica (NS) with various sizes of 12, 20 and 40 nm on the compressive strength and the optimum replacement content of cement mortar containing NS, and then compared this with silica fume (SF). Test results indicated that the inclusion of nano silica considerably improved the compressive strength of cement mortar and cement mortar containing 9% of 40 nm NS gave higher compressive strength compared with NS of 12 nm and 20 nm, due to the agglomeration and ineffective dispersion of the latter. Also, NS gives both pozzolanic activity, packing ability and very small NS particles can participate in the hydration process to generate C-S-H through reaction with Ca(OH)₂.

2.4 Ternary mixture cementitious materials.

A ternary mixture of cementitious materials is simply a mixture of three components, which could be Portland cement, fly ash, slag, silica fume, metakaolin and other natural pozzolans. Nowadays, use of ternary mixtures is becoming common practise to improve the performance of concrete and reduce costs. The reduction in cost is related to the fact that most supplementary cementitious materials are waste industrial materials. Using waste materials decreases the amount of Portland cement that must be manufactured which makes the cement industry more sustainable (Schlorholtz, 2004).

There are a relatively limited number of studies on the fresh and mechanical properties of ternary blended cement concrete. The fluidity of the ternary mixtures varies based on the properties of the materials used. Elahi *et al.*, (2010) examined the workability of ternary blends of Portland cement, GGBS, and silica fume as well as Portland cement, fly ash and silica fume. A water/ cement ratio of 0.3 was used and the superplasticizer was adjusted to achieve equal workability. The result indicated that the superplasticizer needed in the case of the GGBS ternary blend was 2.5 % by mass, and was greater than that needed in the fly ash ternary blend. Zhang and Islam, (2012) evaluated the effect of silica fume and nano-silica (NS) on rate of cement hydration, setting time and strength development of concretes with about 50% fly ash or slag. The results showed that incorporation of nano-silica reduced the setting time and increase the strengths of the slag concrete at these ages, which might be related to the coarse aggregate used which appeared to have reached its strength limit

Nehdi *et al.*, (2004) improved the early age compressive strength of ternary cementitious blends by incorporating 25% GGBS and 25% fly ash when compared to the binary fly ash mix and quarternary mixes with 20% GGBS and 6% RHA or silica fume. Le, (2008) investigated the mix proportions of ultra-high-performance fibre reinforced concrete paving flag (UHPFRC), replacing cement binder by silica fume, slag and fly ash to meet the mechanical performance requirements. The results from this study indicated that the optimum mix proportions obtained in UHPFRC are 10% silica fume and 35% slag by weight of binder. Wongkeo *et al.*, (2014) examined the effect of high calcium fly ash (FA) and silica fume (SF) as a binary and ternary blended cement at high volume replacement on the properties of self-compacting concrete (SCC). The results indicated that the compressive strength decreased by increasing the fly ash content, while at the same replacement level, the compressive strength of SCC increased with increasing silica fume content. Particularly, a mixture of 40%FA and 10%SF at w/b ratio of 0.3 gave high strength self-compacting concrete and equivalent compressive strength to the Portland cement control after 28 days.

2.5 Geopolymer construction materials.

2.5.1 Terminology and Chemistry.

The expression 'Geopolymer' was first used by Davidovits in 1978. Geopolymers are inorganic polymers produced through polymerisation of the silicon (Si) and the aluminum (Al) in source material with an alkaline solution. The chemical synthesis of the geopolymer material is identical to natural zeolitic materials, while the microstructure is amorphous instead of crystalline (Palomo *et al.*, 1999; Xu and van Deventer, 2000). The polymerisation process yields three dimensional polymeric chains and ring structures consisting of a small cation such as Si⁴⁺, or Al³⁺ in a tetrahedral bond (termed polysialate). When SiO₂ and Al₂O₃ coordinate with four oxygen bonds, there must be positive ions (Na⁺, K⁺, Ca²⁺, Mg²⁺, NH⁴⁺) in the framework in order to neutralize the negative charge of Al³⁺ in 4- fold coordination. In general, the principle form pf polysialate geopolymer has the following empirical formula (Davidovits, 1999):

$$Mn[-(SiO_2)z-AlO_2]n.wH_2O$$
(2.1)

Where; M is a cation or alkaline activation agent such as calcium (Ca), sodium (Na) and potassium (K), w is the number of water molecules and n is the degree of polymerization (1, 2, 3, or higher, up to 32) (Davidovits, 2005; Rangan, 2005). Table 2.5 shows a categorisation of geopolymer structures based on the Si/Al atomic ratio;

Category of Polysialate	Molar Ratio of Si/Al	Schematic Structure	Molecular Graphics
Polysialate	1	 M _n -(Si-O-Al-O-) _n 0 0	SIO4 AIO4
Poly- sialatesiloxo	2	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	John Star
Poly- sialatedisiloxo	3	 M _n -(Si-O-Al-O-Si-O-Si-O) _n 0 0 0 0	

Table 2.7: Terminology of poly (Sialate) Geopolymer (Wallah and Rangan, 2006).

Polysilicates are commonly sodium or potassium silicate manufactured commercially by the chemical industry or manufactured fine silica powder produced as a by-product of ferro-silicon metallurgy. Equations (2.2) and (2.3) presents a reaction scheme for the polycondensation process of geopolymerisation from aluminosilicate materials (Davidovits, 1999);

$$n(Si_{2}O_{5},Al_{2}O_{2})+2nSiO_{2}+4nH_{2}O \xrightarrow{NaOH \text{ or } KOH} Na^{+}, K^{+} n(OH)_{3}-Si-O-Al^{-}O-Si(OH)_{3}$$
(Si-Al materials)
$$(OH)_{2} (2.2)$$
(Geopolymer precursor)
$$n(OH)_{3}-Si-O-Al-O-Si-(OH)_{3} + NaOH \text{ or } KOH \longrightarrow (Na+, K+)-(-Si-O-Al-O-Si-O-) + 4nH_{2}O$$

$$(OH)_{2} (2.3)$$

$$(OH)_{2} (2.3)$$

$$(OH)_{2} (2.3)$$

The exact reaction mechanisms for geopolymerisation are not completely clear. Duxson et al., (2007) summarised the steps of geopolymerisation of aluminosilicate material which consist of; (a) dissolution, (b) speciation equilibrium, (c) gelation, (d) reorganization, and (e) polymerization and hardening, as outlined in Figure 2.3. The geopolymerization process occurs entirely through solution with the potential requirements for processing it such as fine material, elevated temperature treatment, etc. The dissolution step starts with an attack to the fly ash particles by alkaline hydrolysis. The dissolution level depends on the properties of the aluminosilicate materials, the concentration of the alkaline solution and the type of alkaline solution (Xu and van Deventer, 2000; Phair and Deventer., 2001; Phair and Van Deventer, 2002). As a consequence, a complex mixture of silicate, aluminate and aluminosilicate species is thereby formed, and the speciation equilibria within these solutions leads to formation of a gel (Swaddle, 2001; Duxson et al., 2007). After gelation the system continues to rearrange and reorganize, as the connectivity of the gel network increases, resulting in the three-dimensional aluminosilicate network commonly attributed to geopolymers.



Figure 2.3: Conceptual models for Geopolymerisation (Duxson et al., 2007).

2.5.2 Geopolymer components.

2.5.2.1 Source Material.

The source material used to produce geopolymers has to contain silicon (Si) and aluminum (Al) oxides in amorphous form. Some natural minerals could be considered as sources for sialate ions such as micas, clays, kaolinite, andalusite, spinel, etc (Davidovits, 1988). Alternatively, industrial waste material such as fly ash, slag, rice husk ash, red mud, silica fume, etc are suitable for producing Geopolymer. This source material might be in single form or in combination with different types of material (Barbosa, 2000; Phair and Van Deventer, 2002). The selection of the proper material for making a specific geopolymer is based on several factors e.g. the cost, availability, specific requirement for the end users and type of application (Wallah and Rangan, 2006).

Between 1972 and 1977 Davidovits developed the first generation of geopolymer technology by using Kaolinate and calcined Kaolinite (metakaolin) as a source of aluminosilicate with alkalis which has been disclosed in various patents issued on the

applications of this so-called "SILIFACE-Process". Later, Davidovits introduced a pure calcined kaolinite called KANDOXI (KAolinite, Nacrite, Dickite OXIde) which is calcined at 750°C for 6 hours. This metakaolin (MK-750) as calcined material performed better geopolymer manufacture compared to natural materials (Davidovits, 2011).

Xu and van Deventer, (2000) conducted an extensive study on a range of aluminosilicate materials which can be used to prepare geopolymer. Their research included sixteen natural minerals which covered the chain, ring, sheet, and framework crystal structure groups, as well as the sodalite, garnet, clay, feldspar, mica, and zeolite mineral groups with the addition of kaolinite to synthesize Geopolymer. These authors established that there are many natural alumino-silicate minerals which provide potential sources for the formation of geopolymer gel, and that it is important to combine natural minerals with kaolinite in order to improve the strength, as a weak structure is formed if only kaolinite is used without presence of other aluminosilicates (Rangan, 2005; Wallah and Rangan, 2006; Ng, 2011). Barbosa et al., (2000) mentioned that calcined source materials such as slag, fly ash and metakaolin yield higher compressive strength than non-calcined materials such as kaolin clay. Metakaolin has interesting properties in producing geopolymer due to its high rate of dissolution in the reactant solution, easier control of the Si/Al ratio and its white colour (Gourley, 2005), but metakaolin is expensive because it has to be calcined at high temperature for several hours and is not available for making geopolymer concrete on a large production scale.

In recent years, research has focused on using industrial by products (fly ash and slag) as a source material for geopolymers. In 2003, Palomo and co-authors investigated various types of fly ash for use as a geopolymer binder. They reported that fly ash to be used as a geopolymer binder should have low calcium content, i.e. less than 5% of unburned material, the iron oxide (Fe₂O₃) content should not exceed 10%, and the reactive silica content (Si₂O₃) should be in the range of 40 to 50%. Also, 80-90% of the fly ash particles size should be smaller than 45 μ m (Fernández-Jiménez and Palomo, 2003; Davidovits, 2011). Gourley, (2003) also showed that the presence of calcium in fly ash in significant quantities could interfere with the polymerisation setting rate and alters the microstructure. This is in agreement with the ASTM C618-

08, (2008) standard which preferred the use of Low Calcium (ASTM Class F) fly ash over High Calcium (ASTM Class C) fly ash as a source material to make geopolymers. However, Van Jaarsveld, (2003) mentioned that higher amounts of CaO resulted in faster strength development and higher compressive strength because of the formation of calcium-aluminate-hydrate and other calcium compounds, especially in the early stages of setting. Further, they reported carbon content, alkali metal content, particle size, amorphous content, and morphology and origin of the fly ash affected the properties of the Geopolymer.

Alkali Activated Slag has been broadly examined and the resulting products are being used in large-scale construction applications in Europe (Glukhovsky, 1994; Van Deventer, 2007). The reaction rate of Alkali Activation Slag depends on many factors such as raw mineral composition, the particle size distribution as well as the concentration and type of the activator (Fernández-Jiménez and Palomo, 2005). Cheng and Chui, (2003) examined granulated blast furnace slag as an active filler for geopolymer production, which achieved a compressive strength of 79 MPa. In addition, these authors found that the setting time of geopolymer correlated well with temperature, potassium hydroxide concentration, metakaolinite and sodium silicate addition. Astutiningsih, (2005) found that the compressive strength of high calcium milled Australian Slag did not exceed 30MPa, although this could be increased by decreasing the water ratio in the sodium silicate solution.

2.5.2.2 Alkaline activator solution.

Any strong alkali solution could be utilized as an alkaline activator for Geopolymer manufacture (Rowles and O'Connor, 2003). The Ancient Egyptians constructed the pyramids by using volcanic ash as source material and lime solution Ca(OH)₂ as an alkaline activator (Davidovits, 2011). According to Glukhovsky *et al.*, (1980), alkaline activators can be classified into six categories based on their chemical composition: (1) Caustic alkalis: MOH; (2) Non-silicate weak acid salts: M₂CO₃, M₂SO₄, M₃PO₄, MF, etc; (3) Silicates: M₂O·nSiO₂ (4) Aluminates:M₂O.nAl₂O₃; (5) Aluminosilicates M₂O·Al₂O₃·(2-6)SiO₂; and (6) Non-silicate strong acid salts: M₂SO₄ (Adam, 2009). Recently, the most widespread alkaline liquid used in geopolymerisation is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate with different Na₂SiO₃/NaOH mass ratios (Barbosa *et al.*,

2000; Xu and van Deventer, 2000; Ryu *et al.*, 2013; Nematollahi and Sanjayan, 2014a). However, both sodium hydroxide and sodium silicate are not available naturally and are produced from energy intensive manufacturing processes. This is particularly the case for sodium silicate, which is produced by fusing a mixture of quartz sand and sodium carbonate at 1350–1450°C, followed by dissolution in an autoclave at 140–160°C under suitable steam pressure (Shi *et al.*, 2005; Davidovits, 2011).

Palomo *et al.*, (1999) found that the types and concentration of alkaline activator play a significant role in the polymerisation process of fly ash based geopolymers, and the combination of potassium or sodium hydroxide with soluble silicate gave compressive strengths around 90 MPa, while in case of alkaline hydroxides alone the strength was between 35 and 40 MPa. This occurs due to the higher reaction rate which occurs with soluble silicate alkaline liquid rather than alkaline hydroxides alone. This was confirmed by Xu and van Deventer, (2000) after extensive research on aluminosilicate material. They demonstrated that increasing the molarities of the alkaline activators improved the reaction rate between the solution and the binder, and that sodium hydroxide solution gives greater dissolution of minerals than potassium hydroxide solution.

Bakharev *et al.*, (1999) examined varying types of alkaline activator for slag based geopolymer concrete, and their compressive strength results indicated that alkali activation slag with liquid sodium silicate (SiO₂/Na₂O ratio of 0.75 and a 4% sodium concentration) provided the best activation compared to sodium hydroxide and sodium carbonate (Chi, 2012). Fernandez-Jimenez, (2006) also explained that the presence of soluble silica in the alkaline activation solution leads to improvements in the mechanical strength, and increases paste density, although this decreases the workability. Schmücker *et al.*, (2005) used fly ash class F as a source material with various alkaline activators and found that NaOH performed better than other activators. Moreover, the presence of water glass with the NaOH alkaline activator improved the strength behaviour of the Geopolymer.

The quantity of alkaline activator solution in the mixture plays an important role on the dissolution of the minerals and mechanical properties of the Geopolymer, and serves as an accelerator of reaction speed by activating Al and Si through a reaction with the binder (Chi, 2012; Nematollahi and Sanjayan, 2014a). The dosage of activator in terms of %Na₂O or %K₂O is defined as the ratio of the oxide content of the alkaline activator to the mass of the binder (Slag or fly ash based geopolymer), whereas the activator modulus (Ms) is the mass ratio of the SiO₂ to the K₂O or Na₂O in the alkaline activator. Krizan and Zivanovic, (2002) noted that the hydration process of alkali-activated slag was influenced by the silica modulus (Ms) and sodium content. Al-Otaibi, (2008) demonstrated that the type of activator and dosage is a crucial factor in Slag based geopolymers with a higher dosage resulting in higher strength and a higher silicate modulus of the activator resulting in higher strength. Sun, (2005) noted that higher NaOH content causes higher dissolution of fly ash binder as more Na+ cations are available for geopolymer.

However, excessive alkalinity has also been suggested to weaken the strength of geopolymer. For example, while Fernandez-Jimenez, (2006) noted that 12.5 molar NaOH produced higher strength than 8 molar NaOH, Palomo *et al.*, (1999) concluded that geopolymer paste with 12 molar KOH has faster activation and higher strength than paste with 18 molar KOH.

2.5.3 Fields of Application.

Geopolymers are a relatively new generation of materials that can be used in a wide range of applications in industry, whether used pure or with fillers or reinforced. Applications are to be found in the automotive and aerospace industries, non-ferrous foundries and metallurgy, civil engineering, cements and concretes, ceramics and plastics industries, waste management, art and decoration, retrofit of buildings, etc. (Davidovits, 1999; Davidovits *et al.*, 2014). Davidovits classified geopolymer properties and application fields based on the Si:Al atomic ratio as explained in Figure 2.4. A low ratio of Si:Al of 1, 2, or 3 initiates a 3D-Network that is very rigid and suitable in the construction material field, while a Si:Al ratio higher than 15 provides a polymeric character to the geopolymeric material (Davidovits, 1999;2011).



Figure 2.4: Chemical structure and applications of geopolymers (Davidovits, 1999).

Geopolymer concrete has been developed for precast structure applications such as concrete sewer pipes, railway sleepers, cemetery crypts, box culverts, and wall panels (Ng, 2011). In 2013, the Wagners EFC Team in Australia tested and supplied 33 large floor geopolymer beams that built the world's first 5 stories of the new Global Change Institute (GCI) building at the University of QLD's St Lucia Campus (Gourley and Johnson, 2005; Gourley, 2014). In 2014, seventy thousand tons of geopolymer concrete were used in building the greenest airport in the world at Brisbane West Wellcamp, Australia, saving more than 6,600 tons of carbon emissions during the construction of the airport.

One of the potential application of geopolymeric materials is in the storage and management of toxic and radioactive wastes, because the behaviour of geopolymer is close to that of zeolitic materials that are known to adsorb toxic chemical wastes (Davidovits, 1988; Wallah and Rangan, 2006). Based on testing, a commercial geopolymer named GEOPOLYMITE 50 manufactured by Cordi-Geopolymere, France is recommended to be used in contaminated waste containment .This is MK-750 Ca based geopolymer prepared by mixing binder slag, silica fume, metakaolin and

mineral fillers with potassium silicate alkaline activator at molar ratio 1.3 (Davidovits, 1988). Between 1994 and 1997, the European multidisciplinary Brite Euram industrial research project GEOCISTEM sought to produce cost effective geopolymeric cements for the long-term containment of hazardous and toxic wastes and for restoring sites highly contaminated with uranium mining waste. Recently, the patented GEOPOLYTECH process is undergoing industrial testing on various sites, applied by B.P.S. Engineering GmbH, Germany.

Geopolymer materials are also used for repairing and retrofitting aging building with fibres. Balaguru *et al.*, (1997) point out that geopolymer provides excellent adhesion to both concrete surfaces and interlaminar fabrics in comparison with organic polymers. Geopolymers can also be applied in high-tech applications such as thermosetting organic resins stable up to 1200 °C (Davidovits, 1988). Geopolymer composite based materials are used in fire resistant aircraft and automotive interiors, e.g. the Benneton F1 team designed a unique thermal shield around the exhaust area of their sports car with dilapsi geopolymer to replace titanium during the Formula One championship in 1994 and 1995. This composite helped the technical team to reduce vibration and engine heat. Nowadays, most F1 teams use geopolymer composite exhaust pipe systems (Davidovits, 2011).



Figure 2.5: Applications of Geopolymer materials (Davidovits, 2011).

2.6 Geopolymer concrete.

2.6.1 Manufacturing of geopolymer.

Geopolymer concrete can be manufactured by following the same techniques used to produce ordinary Portland cement concrete, i.e. dry mixing of the geopolymer binder in a pan mixer, followed by adding liquid components to the solid binder (Davidovits, 2011). Rangan, (2005), who investigated the properties of low calcium fly ash based geopolymer concrete, mixed fly ash with sand for about three minutes then added the liquid phase to the dry material and continued the wet mixing for another four minutes. Liquid components prepared at least one day in advance by mixing sodium hydroxide and sodium silicate were added to the dry materials. Deb *et al.*, (2014) suggest that fly ash, slag and aggregate should be mixed together, followed by addition of the activator solution to the dry materials, and mixing continued for about 5 mins to produced fresh geopolymer concrete.

In general, previous studies show different mixing procedures for producing geopolymer concrete. Cheng and Chui, (2003) suggested mixing KOH with metakaolin for ten minutes, then adding sodium silicate and GGBFS and continuing the mixing process for a further five minutes. Jang *et al.*, (2014) adopted the method of producing fly ash and slag mixtures as follows; fly ash, slag and sodium silicate powder as activator were dry mixed for 2 minutes, then water was added to the mixture and mixed continued for another 2 minutes.

The European research project (GEOASH) (2004-2007) studied variable mixing procedures on fly ash/ slag paste and concluded that the best mixing steps to improve the properties of geopolymer were by mixing potassium silicate solution and slag in different vessels for 20 minutes, then adding these to fly ash powder in a pan mixer and continuing to mix for other 9 minutes, to give a total mixing of 29 minutes. This however is a very complicated mixing operation for scalable industrial use. From an industrial / technical stand point it is preferable to mix first the solid components and then add the potassium silicate solution to the solid blend (Davidovits *et al.*, 2014).

The curing temperature and curing duration have also been reported to play an important role in hardening and in determining the final properties of geopolymer concrete. Previous studies have indicated that the reaction of fly ash geopolymer concrete is slow at ambient temperature and the geopolymerization process improves with various temperatures between 40°C to about 90°C, and curing periods from 1 hour to more than 24 hours (Swanepoel and Strydom, 2002; Chindaprasirt et al., 2005; Rangan, 2005; Görhan and Kürklü, 2014). Hardjito and Rangan, (2005) examined the strength of fly ash based geopolymer concrete and they concluded that compressive strength of the geopolymer improved with increasing curing temperature in the range of 30 °C to 90 °C, although the compressive strength was not significantly increased at temperatures beyond 60 °C. Altan and Erdoğan, (2012) investigated alkali activation slag (AAS) cured at variant temperatures. They found that AAS cured at elevated temperatures can achieve superb compressive strength, considerably higher than conventional Portland cement mortars, very rapidly. AAS mortars cured at ambient temperature have very low early strength, although within a sufficiently long time can attain equal or greater strengths than mortars cured at 80°C. However, van Jaarsveld et al., (2002) stated that elevated temperature curing caused cracking and a negative influence on the properties of the material, and mild curing improved the physical properties of the material.

2.6.2 Development of user-friendly geopolymer.

Geopolymers require chemical addition in order to be used as cementitious materials, and some of the typically applied materials are toxic, and safety procedures are required. Based on the classification of the material safety data sheets, the geopolymeric alkaline elements can be ordered in two categories: (a) corrosive and (b) irritant (user friendly) products as shown in Table 2.6.

Corrosive	Irritant	
CaO (quick lime)	Ca(OH) ₂	
NaOH	Portland cement	
КОН	Iron slag	
Sodium metasilicate	Slurry soluble silicate/kaoline	
SiO ₂ :Na ₂ O=1	MR 1.25 <sio<sub>2:M₂O<1.45</sio<sub>	
Any soluble silicate	Any soluble silicate	
MR SiO ₂ :M ₂ O<1.45	MR SiO ₂ :M ₂ O>1.45	

Table 2.8: Corrosive and irritant chemicals (Davidovits, 2011).

Many of the previous studies on geopolymer concrete have focused on material properties, and have not considered end user safety. For example, Nicholas and Fletcher, 2005 used alkaline activator MR SiO₂:M₂O of 0.3 with fly ash based geopolymer. Skvara and kastanek, 2003 utilised MR ratios ranging between 0.4 and 1. Palomo *et al.*, (1999), Jin *et al.*, (2012), Ismail *et al.*, (2013) and Ismail *et al.*, (2014) suggest the use of sodium metasilicate MR=1 which is available commercially. Others have used even stronger alkaline activators, like Astutiningsih, (2005) who keep the MR in the range of 0.6. Based on the safety classification, all of these alkaline activator conditions are corrosive to strongly corrosive and safety procedure must be followed such as handling with gloves, glasses and masks (Davidovits, 2011). In 2003, Davidovits presented a user-friendly system by using SiO₂:M₂O alkaline activator in the range of 1.25-2.0. A newer geopolymerisation method also produced fly ash based geopolymer concrete in a user friendly system by using geopolymeric slurry including K-silicate solution (molar SiO₂:K₂O>1.4), slag and water (Davidovits *et al.*, 2014).

2.6.3 Fresh properties of geopolymer mortar/ concrete.

Fresh state behaviour occurs immediately after mixing of the geopolymer concrete. Physically, the initial geopolymer binder is a fluid resin that changes into a fluid-like paste, which over time sets and hardens into a hard highly resistant material. Setting time or working time of geopolymer concrete is not characterised well in the literature, and there are limited publications on this topic. Geopolymer setting can be achieved within minutes to up to 4 hours, as there are many factors which effect the setting time, such as curing temperature and the chemical composition of the source material. Davidovits, (1999) mentioned that metakaolin MK750 can set in a short time at room temperature. Wang *et al.*, (1995) mentioned that alkali activated slag geopolymer starts to harden in 15 minutes when producing > 70 MPa ultimate compressive strength without using admixtures. Cheng and Chui, (2003) stated that the setting time for GGBS based geopolymer paste was between 15 to 45 minutes at 60°C curing temperature. In contrast, Rangan, (2005) mentioned that fly ash based geopolymer concrete takes more than 24 hours to set at low temperature due to the slow rate of chemical reaction.

Recently, researchers have attempted to improve the mechanical properties and setting time of fly ash geopolymer concrete by inclusion of slag as a source of calcium (CaO)

(Lee and Lee, 2013; Deb *et al.*, 2014; Jang *et al.*, 2014). Nath and Sarker, (2014) noted that the initial and final setting time of fly ash geopolymer decreased with increasing slag content. They found that the initial setting time with 10% slag in the binder (fly ash and slag) was 290 minutes, which decreased to 94 and 41 minutes on inclusion of 20% and 30% slag/ binder ratio respectively. In addition, water content and alkaline activator types and content impact on the setting time of geopolymer concrete. Lee and Lee, (2013) examined the setting time of fly ash/ slag based geopolymer paste with varying NaOH molarities (4, 6 and 8 M) and waterglass/ NaOH ratios. They reported that the setting time of geopolymer decreased as the amounts of slag and water glass and the molarity of the NaOH solution increased.

Nath and Sarker, (2014) mentioned that fly ash/ slag geopolymer paste needs a longer time to set when alkaline solution content is increased from 35% to 40% and 45% in the mixture, due to higher water to solid ratio and deceleration of the condensation process of geopolymerisation. Also, these authors found that decreasing the sodium silicate to sodium hydroxide ratio of the alkaline solution with a constant amount of alkaline activator solution cause increased the setting time. This is due to the reduced Si/Al ratio in the mix. As the amount of Si decreases in the paste, polymerisation process slow down to some extent.

There have been attempts to control the setting time of geopolymer concrete especially for mixtures which have rapid setting time, and to avoid the complication caused by long delays in the working time between mixing and placing on the matrix. Retarding admixtures are mainly based on material having lignosulfonic acids, hydroxy-carboxylic acid, tatric acid, citric acid, and their salts etc., and the optimal dosage is in the range of 1 to 1.5% (Davidovits, 2011). Retarding admixtures that are known to delay the setting time of Portland cement paste have no effect on alkali activation slag due to the different chemical composition of the slag cements (A1-Otaibi, 2002). Marta *et al.*, (2008) stated that the setting time of slag based geopolymer could be enhanced up to 180 min with the use of a naphthalene based admixture and extended mixing time. Jang *et al.*, (2014) argued however that the initial and final setting time of fly ash/ slag based geopolymer paste was relatively unaffected by the presence of the naphthalene-based superplasticizer. Polycarboxylate-based superplasticizer at 4% addition has been observed to retard the initial and final setting time by 50 min and 70

min, respectively (Jang *et al.*, 2014). Ahmed *et al.*, (2012) and Fadhil *et al.*, (2011) prepared fly ash self-compacting concrete (SCC) where sodium silicate and sodium hydroxide solution was employed as the alkaline activator. Different dosages of superplasticizer of 3%, 4%, 5%, 6% and 7% were used, and results of slump flow tests showed higher flow with higher dosage of superplasticizer.

2.6.4 Hardened properties of geopolymer mortar/concrete.

2.6.4.1 Mechanical properties of geopolymer mortar/concrete.

Geopolymer concrete cured under elevated temperature has excellent mechanical properties comparable to Portland cement concrete, and high early strength (Lee and Lee, 2013; Nath and Sarker, 2014). Previous research indicates that the chemical reactions and strength development of geopolymer concrete are affected by number of parameters such as chemical activator composition and dosage, raw materials, and curing conditions (Duxson, 2007; Ryu et al., 2013; Deb et al., 2014). Goretta, (2004) measured the compressive strength of geopolymer derived from class C fly ash, slag and sodium silicate as alkaline activator and they reported that compressive strengths of 35 MPa could be obtained at 14 days. Shen et al., (2011) examined the flexural strength and compressive strength of alkali-activated FA/ slag cement with different fly ash to slag levels (0%, 30%, 60%, 50%, 40%, 70% and 100%, by weight). The results indicated that mechanical strength increased as the slag content increased. Similarly, Rashad and Khalil, (2013) found that the compressive strength, splitting tensile strength and flexural strength improved by increasing the slag content in alkaliactivated fly ash/slag geopolymer concretes. On the other hand, fly ash geopolymer concrete has lower Young's modulus than that of OPC concrete (Rangan, 2005; Fernandez-Jimenez, 2006).

Escalante *et al.*, (2006) examined the effect of different slag to fly ash wt. ratios (100 %, 75 %, 50 %, 25 % and 0 %) on compressive strength. Sodium silicate with modulus (SiO₂/Na₂O) of 0, 0.75, 1, 1.5 and 2 was used as alkaline activator. The %Na₂O was added at 4%, 6% and 8%, related to the binder weight. The pastes were cured at 75 °C for 24 hrs and then at 20 °C up to 28 days. Their results indicated that 100 % slag materials with 4% Na₂O had the highest strength (80–85 MPa); the optimum modulus was 1.5. In contrast, 100% fly ash based geopolymer showed the lowest compressive strength values at around 25 MPa. The highest strength for the composite containing

75 % slag were 56–60 MPa at 4 % Na₂O and modulus 1 and 1.5. For 50 % slag composite paste, the strengths were 45–48 MPa at 4 % Na₂O and the optimum modulus was 1–1.5. For 25 % slag composite paste, the strength reached 30–35 MPa at 4 % Na₂O and modulus 1.5.

Wang *et al.*, (2012) examined the compressive strength of alkali-activated FA, slag and MK cementitious materials prepared by a hydrothermal method. Sodium silicate with modulus ca. 1 was used as the alkaline-activator. Compressive strength results indicated that this material had higher mechanical strength and the best strength value reached nearly 80 MPa. The authors suggested that this was due to the slag content in the mixture, which generated more hydration products of CSH and hydrated calcium aluminates. Yip *et al.*, (2005) replaced slag with metakaolin at different wt. ratios 0%, 20%, 40%, 60%, 80% and 100%. Sodium hydroxide and sodium silicate were used as alkaline activator solution with two different molar ratios of 2 and 1.2. All specimens were cured under elevated temperature of about 40 °C for 24 hrs and then left under 20 °C ambient temperature up to the testing date. Compressive strength results showed that the mixtures containing 20% metakaolin to slag wt. ratio gave the highest strength in both molar ratios.

Cahit and Duran, (2012) examined the mechanical properties of alkali activated slag mortars activated with sodium silicate. The slag was partially replaced with Portland cement (wt. ratios 0%, 20%, 40%, 80% and 100%) and three different Na dosages were used. Their results showed that the compressive strength and flexural strength decreased as the Portland cement amount increased and that the optimum strength was achieved from alkali activated slag without any replacement of Portland cement. Guo *et al.*, (2010) partially replaced fly ash by Portland cement at levels of 0%, 10%, 20%, 30%, 40% and 50%, by weight. Sodium hydroxide and sodium silicate were used as alkaline activators and the specimens were cured at 23 °C. Their results showed that 40% Portland cement gave the best compressive strength compared to the other mixture ratios. Lohani *et al.*, (2012) studied the workability and compressive strength of fly ash/ Portland cement based geopolymer activated with sodium hydroxide solution as alkaline activator. Fly ash was partially replaced with Portland cement at levels of 0%, 10%, 25%, 40%, 60% and 100%, by weight. Their results showed that increasing the Portland cement content enhanced the compressive strength and

reduced the workability. Palomo *et al.*, (2007) examined the effect of alkaline activator type on the compressive strength of 70 % Fly ash and 30 % Portland cement clinker. The composite was activated with either sodium hydroxide solution or waterglass and sodium hydroxide solution. A mixture hydrated with deionised water was employed for comparison. The compressive strength results indicated that the highest compressive strength was achieved when waterglass with sodium hydroxide solution was utilized as the alkaline activator, followed by the mixture mixed with deionised water, and then the mixture activated with sodium hydroxide solution alone.

Rashad and Khalil, (2013) studied the compressive strength of alkali-activated slag geopolymer with partial replacement by silica fume at different levels, with sodium silicate solution activator and curing under room temperature. The authors found that 5% of silica fume replacement slag gave the highest compressive strength at ages of 7 and 28 days. Serdar, (2013) examined the effect of silica fume to slag wt. ratio (0%, 10% and 20%) in geopolymer activated with sodium hydroxide and sodium silicate and cured at 20 °C and 90% RH for 5 hrs, then in steam at 70 °C for 6 hrs. The results indicated that 10% silica fume improved the compressive strength by 4.24%, while 20% silica fume reduced the compressive strength by 15.42%. On the other hand, the flexural strength decreased by 29.59% and 32.65% with the inclusion of 10% and 20% silica fume, respectively. Douglas and Brandstetr, (1990) stated that the compressive strength of alkali-activated mortars activated with sodium silicate and modified with 8% Silica fume and 2% lime reduced the compressive strength at the early age of 1 day, but increased the compressive strength at ages of 7 and 28 days, compared to the respective control activated slag mortar without modification. Debabrata et al., (2010) and Suresh et al., (2011) replaced fly ash with silica fume at levels 0%, 2.5% and 5%, by weight in mortar and pastes. Sodium silicate and sodium hydroxide was utilized as activator. The alkaline activator concentration was Na₂O and molar ratio was 1. All the specimens were cured under 85 °C for 48 hrs then left to cool inside the oven. The results showed that compressive strength was increased in the mortar as the silica fume content increased. On the other hand, the compressive strength of the geopolymer paste decreased with increasing silica fume content. Smith et al., (2011) examined different replacement wt. percentages of fly ash with silica fume at levels of 0%, 10%, 20%, 30% and 40%. Two sodium hydroxide concertation 10M and 18M were examined, at sodium silicate to sodium hydroxide ratio was 2.5. Their results showed that compressive strength and bonding strength increased with increasing silica fume content and sodium hydroxide concentration.

2.6.4.2 Durability characteristics of geopolymer mortar/ concrete.

One of the main problems associated with OPC concrete is its long-term durability, which had always been an issue in physically and chemically aggressive environments (Wallah and Rangan, 2006; Singh et al., 2015b). The deterioration of concrete is generally evaluated for sulphate attack, atmospheric carbonation, chloride induced corrosion, drying shrinkage, freeze-thaw attack and alkali-silica reaction (Singh et al., 2015a). The durability properties of geopolymer paste/ concrete have been studied by several authors (Singh et al., 2015b). Previous studies have reported that geopolymer concrete possesses good durability properties such as freeze-thaw resistance, sulphate resistance, corrosion resistance, acid resistance, fire resistance, and no dangerous alkali-aggregate reaction (Wallah and Rangan, 2006). Geopolymer concrete has been found to be more durable than OPC concrete in term of heat and fire resistance. Davidovits, (1999) noted that geopolymer concrete is stable up to 600 °C, whereas OPC concrete shows a rapid deterioration in compressive strength at 300 °C. Kong et al., (2008) mentioned that the compressive strength of fly ash based geopolymer pastes increased by 6% after exposure at 800 °C, while the strength of metakaolin-based geopolymer pastes was reduced by 34%. Providing sufficient workability for OPC concrete demands the presence of water in the mixture which causes high porosity and results in cracking, which leads to reduced durability of the concrete matrix. Geopolymer materials do not require the addition of water in their manufacture and have a much lower shrinkage than OPC concrete (Wallah and Rangan, 2006). Maurice and Sanajayan, (2010) examined the effect of elevated temperature up to 800 C, for 1 hour, on the mechanical performance of fly ash and slag paste. A combination of sodium silicate and 8M of sodium hydroxide was used as the alkaline activator and slag was replaced by fly ash at levels of 0%, 50%, 65% and 100%, by weight. Results showed that specimens with very low strengths (<7.6 MPa) experienced an increase in residual strength of up to 90%. Specimens with initial strengths of 28 MPa had residual strength losses of around 70%, while geopolymer specimens with higher initial strengths approaching 83 MPa had residual strength losses of approximately 90% after exposure to 800 °C. Cheng and Chui, (2003) examined the fire resistance of a 10 mm thick panel of slag/ metakaolin geopolymer exposed to a 1100 °C flame.

The measured reverse-side temperature reached 240–283 °C after 35 min. They found that the fire resistance might be enhanced by the KOH or the alkali concentration and metakaolin content.

Several attempts have been made to study the chemical resistance of geopolymer concrete (Hardjito, 2004) .Geopolymeric material tends to show chemical resistance higher than OPC concrete. The better performance of geopolymer cement in term of acidic environments could be attributed to the lower calcium content of the source material, as geopolymer concrete does not produce lime (CaO) during its hydration process, and is not dissolved by acidic solutions unlike Portland cement concrete (Sanni and Khadiranaikar, 2012; Shaikh, 2013a). Temuujin J et al., (2011) observed that the acid and alkaline resistance of fly ash based geopolymer depends on its minerologyical composition. Wallah and Rangan, (2006) mentioned that the compressive strength of fly ash based geopolymer was reduced by 20% after 12 months exposure to 0.5% H₂SiO₄ solution. This value was ~52% and ~65% respectively when samples were exposed to 1% and 2% H₂SiO₄ solution. Similarly, Bakharev, (2005c) exposed slag based geopolymer and OPC concrete to acetic acid solution (pH 4) for 12 months. Their results exhibited a 33% reduction in the strength of the slag-based material while the reduction in OPC strength was 47%. The slag particles and low calcium C-S-H products with average Ca/Si ratio of 1 were more stable in the acid solution than the constituents of the OPC pastes.

Hardjito, (2004) found that there was no considerable effect of 5% sodium sulfate (Na₂SiO₄) solution on the compressive strength, the dimensions and weight loss of fly ash based geopolymer after 3 months exposure. Rajamane *et al.*, (2012) found that exposing fly ash based geopolymer concrete to 5% Na₂SO₄ and 5% MgSO₄ for 3 months showed marginally less strength loss than in OPC concrete. The compressive strength was reduced by 2-29% and 9-38% in the fly ash based geopolymer and OPC concrete, respectively. This reduction in the strength of OPC concrete could be explained due to formation of expansive gypsum and ettringite which can cause expansion, cracking and spalling in the concrete. In contrast, geopolymer concrete does not contain Ca(OH)₂ and mono-sulphoaluminate in the matrix to cause expansion (Singh *et al.*, 2015a). Idawati *et al.*, (2012) reported the behaviour of fly ash/slag based geopolymer binder exposed to different forms of sulfate salts (5 wt% sodium sulfate

(Na₂SO₄) or 5% magnisum sulfate (MgSO₄)) solutions) for 3 months. Their results showed that immersion of fly ash/slag based geopolymer in sodium sulphate did not lead to any apparent degredation of the binder while immersion in magnesium sulphate caused degradation of the binder system. The products of magnesium sulfate attack were poorly cohesive and expansive, leading to dimensional instability and loss of mechanical performance.

Adam, (2009) studied water sorptivity, chloride and carbonation resistance of fly ash and slag based geopolymer concretes. Results from this study indicate that fly ash based geopolymer concrete performed better than OPC and alkali activated slag concrete in water sorptivity and chloride penetration. Muntingh, (2006) indicated that the chloride diffusion rate of geopolymer concrete was more than 300 times lower than that of Portland cement concrete. Olivia and Nikraz, (2012) mentioned the lower permeability of geopolymer concrete with alkaline activator to fly ash ratio, 0.30–0.40 and cured at 60 °C for 24 hrs than OPC concrete due to its denser paste and smaller pore inter-connectivity. Chi and Huang, (2013) reported the percentages of water absorption of geopolymer mortar made of different material combinations of fly ash and slag wt. ratios of 100%, 70%, 50%, 30% and 0%. Two concentrations of sodium oxide (Na₂O) of 4% and 6% by cementitious weight were examined and sodium silicate with modulus ratio of 1 was utilized as alkaline activator. Their outcomes indicated a reduction in the percentage of water absorption with increasing slag content at both Na₂O concentrations. Jin et al., (2012) examined the porosity of alkali activated slag, fly ash and metakaolin materials produced by a hydrothermal method. Sodium silicate was utilized as alkaline activator with the modulus adjusted to 1.0 by dissolving sodium hydroxide, and the ratio of water to solid binder was approximately 0.35. Their results showed that the porosity was less than 36% after the hydrothermal process due to the effect of these materials in compacting the structure. Bernal et al., (2012) examined the capillary sorptivity, rapid chloride permeability test (RCPT) and water absorption of alkali activated slag/ metakaolin concrete. The ratios of slag to total binder were 0.8, 0.9 and 1, and a combination of sodium silicate and sodium hydroxide used as alkaline activator to reach the overall molar ratios (SiO₂/Al₂O₃) of 3.6, 4.0 and 4.4. The authors reported that increasing metakaolin content and alkaline activator concentrations reduced water absorption and water sorptivity and gave lower chloride permeability.

Shrinkage is the reduction in volume at constant temperature without external loading. It is a vital material characteristic that considerable effects long-term behaviour of designed structures (Gholami *et al.*, 2013; Rashad, 2014b). Much more research has been carried out on the shrinkage behaviour of alkali activated fly ash and alkali activated slag compared to fly ash and slag based geopolymer. According to previous studies (Hardjito and Rangan, 2005; Wallah and Rangan, 2006; Wallah, 2009), fly ash based geopolymers show relatively low shrinkage and have good mechanical properties compared to ordinary Portland cement (OPC). However, these require elevated curing temperatures to achieve high strength and low shrinkage, which may result in considerable energy consumption. In contrast, slag based geopolymer has been shown to exhibit a higher rate of shrinkage compared with OPC (Douglas *et al.*, 1992; Wang *et al.*, 1995; Lee *et al.*, 2014).

Fernandez-Jimenez, (2006) also measured significantly lower of drying shrinake of fly ash based geopolymer concrete up to 90 days of age. In their study, geopolymer concrete specimens were cured at 85 °C for 20 hours. Collins and Sanjayan, (1999) mentioned a relatively high amount of drying shrinkage (around 1500 microstrain) for ambient cured slag based geopolymer concrete when compared to Portland cement concrete (around 700 microstrain) of the same grade (Neupane, 2016). Chi and Huang, (2013) measured the drying shrinkage of fly ash/ slag based geopolymer at 7, 14 and 28 days. Different mixtures of fly ash to slag wt. ratios of 100%, 70%, 50%, 30% and 0% were examined and the results reported that the drying shrinkage increased as the slag content increased. Similarly, Rashad, (2013) evaluated the effect of slag content on drying shrinkage of fly ash based geopolymer up to 91 days. A combination of sodium silicate and sodium hydroxide was utilized as the alkaline activator. The results showed that the drying shrinkage increased as the slag content increased. Serdar, (2013) measured the drying shrinkage of geopolymer mortar prepared from different combinations of FA/slag ratios of 40/60, 20/80, 0/100, by weight, with NaOH. The specimens were cured under steam at 70 °C for 6 hours. The drying shrinkage results up to 4 months showed an increase in the drying shrinkage as slag content increased.

2.7 Fibre reinforced concrete.

Fibre reinforced concrete (FRC) may be defined as a composite material made with Portland cement, aggregate, and incorporating discrete discontinuous fibres. Although concrete is the most used construction material in the world, this material has a still some serious problems associated with its use in some applications. For example, normal or high strength concrete have good compressive strength properties but a very low tensile strength, and usually fail by sudden propagation of cracks due to their brittle nature under tensile stress. In order to solve brittle failure, an appropriate load carrying mechanism must be provided across the cracks such as fibres reinforcement (Sebaibi *et al.*, 2014). Experimental studies have shown that the addition of fibres develops the structural properties of concrete such as flexural strength, compressive strength, tensile strength, creep behaviour, impact resistance, ductility and flexural toughness (Topçu and Canbaz, 2007; Atiş and Karahan, 2009). Also, the durability properties of fibre reinforced cement concrete are improved by bridging of cracks (Bernal *et al.*, 2010; Shaikh, 2013b).

Fibre reinforced concrete (FRC) has been widely used in many applications and various types of fibres have been utilized. Generally, glass fibres are used in production of thin sheet elements for instant precast architectural panels because of their capacity to produce relatively light weight and thin sections. Synthetic fibres, such as polyethylene, are used to enhance resistance to cracking caused by drying shrinkage. For structural performance, steel fibres have competitive advantages over other fibres due to their high elastic modulus and ability to form a strong bond with the surrounding cementitious matrix. For example, industrial pavements, non-structural precast elements (pipes, culverts and other small components) and fibre reinforcement is particularly appealing for large structural elements (Soutsos *et al.*, 2012).

The mechanical performance of fibre reinforced cementitious composites depends on the material parameters (both of the fibres themselves, and the cementitious matrix). These parameters include strength, aspect ratio, Poisson's ratio and shape of fibres; the stiffness, strength, and shrinkage of the matrix; and frictional bond properties and the physio-chemical interactions at the interface between the cementitious matrix and the fibres. The type and the quantity of fibres influences the mechanical and/or
chemical properties as well as the extent that the fibres influence the matrix properties. The fibres usually used in concretes are divided into two broad categories. The first category is low modulus, high elongation fibres such as nylon, polypropylene and polyethylene in which the fibres enhance primarily the energy absorption characteristics only. The other category of fibres is high strength, high modulus fibres such as steel, glass and asbestos in which the fibres enhance the strength as well as the toughness of the composites (Swamy, 1992). While considerable advances have been made in fibre reinforced concretes, efforts have also been made to replace the cement based binder in current FRCC with "geopolymeric binder", resulting in fibre reinforced geopolymer composites (FRGCs).

2.7.1 Factors influencing the interfacial bond of fibre reinforced concrete.

The mechanical behaviour of fibre reinforced concrete is considerably influenced by the types of fibres used, and the matrix composition.

2.7.1.1 Fibre Types.

Fibers of different materials and geometric properties are used in construction applications, which can be divided into two major categories of high modulus (metallic) and low modulus (non-metallic) fibres (Jiang, 2003). Each category enhances particular properties of the matrix. Generally, incorporation of metallic fibre result in flexural strength enhancement due to their higher stiffness while non-metallic fibres control the plastic shrinkage of the matrices since they have a higher aspect ratio and surface contact area. In this present investigation, steel, PVA and glass fibres were selected for the development and investigation of the structural behaviour of geopolymer concrete. The following sections review and discuss the behaviour and constitutive laws of steel fibre reinforced concrete.

2.7.1.1.1 Steel fibre reinforced concrete.

Steel reinforcing fibres are commonly manufactured from carbon steel or stainless steel. Steel fibres are preferred over other fibres for structural performance because of their high elastic modulus around 200 GPa, tensile strength ranging from 245 to 2300 MPa and ability to form a strong bond with the surrounding cementitious matrix (Ng, 2011). Steel fibre reinforced concrete (SFRC) is increasingly being used in structures such as flooring, housing, precast, tunnelling, heavy duty pavement and mining.

Generally, steel fibre varies in length up to about 60 mm, aspect ratios vary from 20 and 100, and fibres have different cross sections in order to gain pull-out resistance, have enlarged, flattened or hooked ends, roughened surface textures or wavy profiles as shown in Figure 2.6 (Atiş and Karahan, 2009). The most suitable volume fraction values for concrete mixes are between 0.5% and 2.5% by volume of concrete (Yazıcı, 2007).

At the beginning of macrocracking, the opening and growth of cracks is controlled by the bond resistance of the fibre. Types of fibre matrix bonds are by chemical adhesion, friction and bonding due to mechanical anchorage induced by deformation. However, the adhesion bond component is comparatively weak especially in the case of traditional fibre reinforced concrete where the typical fibres used are smooth, and the effective bond cannot be improved. Here, the bond is developed through mechanical anchorage (e.g. end-hooks and fibre bending or snubbing) and the frictional bonds are significant (Htut and Foster, 2008; Htut, 2010; Abdallah *et al.*, 2016).



Figure 2.6: Types of steel fibre (Dinh, 2009).

Soutsos *et al.*, (2012) investigated the influence of shape, aspect ratio and dosage of steel and synthetic fibres on the flexural performance and other mechanical characteristics of fibre reinforced concrete. Results indicated that the inclusion of steel fibres at dosage rates of 30 kg/m³ and 50 kg/m³ increased the compressive strength by about 4 and 5MPa. Addition of synthetic fibres for dosage rates of 4.5–5.3 kg/m³ slightly increased the compressive strength to about 2-3 MPa. Topçu and Canbaz, (2007) examined the effects of addition of steel and polypropylene fibre on the

mechanical properties of cement containing various percentages of fly ash. The results indicated that steel fibre reinforced concrete showed better compressive strength and splitting tensile strength than polypropylene fibre reinforced concrete. Ahmed *et al.*, (2007) presented the results of an experimental investigation on the strain-hardening behaviour of hybrid steel–polyethylene (PE) fibre reinforced cement composites containing a high volume of fly ash under four-point bending. A comparison with hybrid steel– polyvinyl alcohol (PVA) fiber composites was also presented. Hybrid steel–PE fibre composites showed lower ultimate strength but higher deflection capacity at the peak load than hybrid steel–PVA fiber composites. A hybrid combination of 1.5% steel and 1.0% PVA exhibited the best performance in terms of highest flexural strength, while 0.5% steel and 2.0% PE exhibited highest deflection and energy absorption capacities.

In the literature, there are few studies focusing on the effect of fibres on the mechanical properties of geopolymer concrete. Bernal *et al.*, (2010) examined the mechanical properties of added steel fibres in alkali activation slag concrete with waterglass. Their results indicated that the splitting tensile and flexural strengths significantly improved, while a reduction in compressive strength with increasing steel fibre amounts from 40 kg/m³ to 120 kg/m³ was observed. Aydın and Baradan, (2013) examined the influence of length and volume fraction of steel fibres on the mechanical and drying shrinkage behaviour of steel fibre reinforced alkali-activated slag/silica fume (SF) mortars. The composite ratio of slag/SF was 80/20 with two different aspect ratio of steel fibre (37.5 and 81.25) and four different dosage of 0.5%, 1.0%, 1.5% and 2.0% were utilized. The results indicated that increasing the steel fibre content lead to a reduction in the workability and drying shrinkage. Compressive strength and flexural strength as well as toughness clearly developed with an increase in fibre contents and fibre length.

2.7.1.1.2 Polyvinyl alcohol (PVA) fibre reinforced concrete.

Amongst polymeric fibres, PVA fibre is a comparatively new fibre and is used in concrete to resist alkaline environments (Garcia and Antoine, 1997). PVA fibres have high tensile strength (approximately 900 MPa), a low modulus of elasticity (around 29 GPa), and a hydrophilic surface which creates a strong chemical bond with cementitious material. After matrix cracking, the high tensile strength of the fibre experiences the first crack stress and effectively resists the pulling out load due to its

strong bond with the matrix. The low modulus of the fibre helps to transfer the load to other parts of the material, distributing load over the whole loading surface. Moreover, the cost of PVA fibre is cheaper than that of high elastic modulus polyethylene fibre and steel fibre on an equal volume basis (Li *et al.*, 2001; Jiang, 2003; Pan *et al.*, 2015). PVA fibre is therefore considered as an alternative reinforcement to attain strain hardening cementitious materials (Jiang, 2003).

Recently, polyvinyl alcohol (PVA) fibre use has been investigated in concrete and these fibres have produced excellent results (Zhang Y, 2009; Shafiq *et al.*, 2016). Garcia and Antoine, (1997) investigated the use of two PVA fibre lengths (4 mm and 12 mm) as a reinforcement in cement based matrices. A fibre volume fraction of 2% was used and a three-point bending test was performed. The results showed that inclusion of PVA fibre in cementitious matrices led to multiple cracking and pseudo-strain hardening behaviour. Increasing the fibre length resulted in greater strain hardening capacity. Li *et al.*, (2001) studied the strain hardening performance of fibre reinforced mortar containing 2% PVA fibre with a surface oil coating. All specimens showed clear pseudo strain hardening behaviour. The strong chemical bond between the cementitious matrix and the oiled PVA fibres led to the rupture of bridging fibres rather than their pullout during the opening of a matrix crack. Therefore, a fibre surface oiling was applied to weaken the bond.

Natali *et al.*, (2011) examined the flexural behaviour of a metakaolin/slag based fibre reinforced geopolymer concrete (FRGC) containing four different types of fibre (carbon, E-glass, polyvinyl alcohol (PVA) and polyvinyl chloride (PVC)). All fibres showed improvement in the flexural strength of FRGC, with greatest improvements in carbon and PVA fibre reinforced FRGC which exhibited a 50% and 62% increase in flexural strength, respectively along with significant improvement in post-crack ductility. Puertas *et al.*, (2003) examined alkali activated fly ash with polypropylene fibres at levels of 0%, 0.5% and 1%, by mortar volume. Their results indicated that higher compressive and flexural strengths occurred at an age of 2 days in AAFA mortar with an increase in fibre content, whilst lower compressive and flexural strengths were obtained at an age of 28 days. Lee *et al.*, (2012) presented a feasibility study of strain-hardening fibre reinforced cementless composite using alkali activated

slag (AAS) based mortar and polyvinyl alcohol (PVA) fibre. Test results showed the feasibility of attaining tensile strain up to 4.7% in fibre reinforced AAS composites, compared to 0.02% for the mortar matrix alone.

2.7.1.2 Matrix composition and its mechanical properties.

The bond behaviour between discontinuous fibre and the surrounding cementitious matrix considerably effect by the mechanical properties of the cementitious material. In OPC concrete, the presence of fine cementitious materials (for instance finely ground cement, slag, fly ash, silica fume, latex and metakaolin) will develop the particle packing of the cementitious matrix and enhance the interfacial microstructural of the concrete and, consequently, develop the frictional bond strength and increase debonding energy (Banthia, 1990; Naaman and Najm, 1991; Najm *et al.*, 1994; Banthia and Trottier, 1995; Guerrero and Naaman, 2000; Ivorra *et al.*, 2010).

Naaman and Najm, (1991) noted that the interfacial bond characteristics between fibre and the matrix are very complex due to the combined actions of several bond components, with one of them being the strength characteristics of the matrix. Their experimental results indicated that higher bond strength could be observed between the fibre and the matrix as the compressive strength of the matrix increases. Similarly, Khaloo and Kim, (1997) concluded that an increase of concrete matrix strength will result in stronger bond development at the fibre/concrete interface and, consequently, FRC specimens have a higher ultimate shear stress than their plain concrete counterparts. Ultra-High Performance Fibre Reinforced Concrete (UHPFRC) is a relatively new construction material, which is produced by a combination of fibre with high strength concrete matrix using high binder dosage and low water to binder ratio (Yu *et al.*, 2015b).

2.7.2 Strain Hardening performance of FRCC.

Over the last several decades, considerable efforts have been made to improve the behaviour of cement based materials by involving discontinuous fibres as a reinforcement, resulting in new types of fibre reinforced concrete materials (Yun, 2013a; Nematollahi *et al.*, 2014;2015). Strain hardening cementitious composites (SHCC), also known as engineering cementitious composite (ECCs), are new classes of high performance fibre reinforced cementitious composites developed by (Li and

Leung, 1992; Li *et al.*, 1993; Li, 1998), tailored using micromechanics theory, which exhibit high ductility and durability, and self-controlled micro-crack width (Khan and Abbass, 2016). ECC consists of fine sand, mineral admixture, cement, water, admixtures which are used to enhance the strength and workability, and less than 2% volume of short fibres. ECC exhibits multiple cracks formed uniformly over the length of the specimen, and the opening of each crack is usually controlled to be less than 100 μ m, subsequently, the ultimate tensile strain can reach over 2.0% (Pan *et al.*, 2015). SHCC materials have good characteristics for exhibiting multiple cracking under flexural and direct tensile loading rather than single localized cracking at the failure plane (Yun, 2013a).

Li, (1998) initially produced ECC with high-modulus polyethylene (PE) fibre, then, Kanda and Li, (1998) used more environmentally friendly polyvinyl alcohol (PVA) fibres with excellent alkali resistance to produce ECC (PVA-ECC). The PVA fibre has higher tensile strength and elastic modulus than polypropylene (PP) fibre. As noted previously, unoiled PVA fibre could be ruptured in a cementitious matrix because of the strong chemical bonding to cement hydrates; thus, the interface is engineered by applying an oil coating to the surface of fibre to decrease the bond. The oiling content is 1.2% by fibre weight. Recently, oiled PVA fibre is commonly used in ECC (Huang X *et al.*, 2013; Zhang *et al.*, 2014).

Engineering cementitious composites (ECC) can considerably contribute to enhancing the service life of civil infrastructure. Unlike traditional tension softening concrete and fibre-reinforced concrete (FRC), ECC exhibits metal-like tensile strain-hardening behaviour after matrix first cracking. Figure 2.7 shows the typical tensile stress-strain curve of ECC. Pseudo strain hardening behaviour, i.e., a post-cracking strength larger than the first cracking strength, is generally accompanied by multiple cracking and a related large energy absorption capacity. The tensile ductility of ECC is several hundred times that of normal concrete and the fracture toughness of ECC is similar to that of aluminum alloy (Yang *et al.*, 2007; Yun, 2013b).



Figure 2.7. Typical tensile stress-strain curve of ECC (Tran et al., 2015).

The strain hardening concrete composite as repair material shown superior durability and mechanical characteristics due to the high compatibility of their deformation with existing concrete, tensile ductility, and self-controlled micro-crack width (Kamal et al., 2008). The performance of SHCCs for seismic and non-seismic structural applications has been assessed via various experiments (Choi et al., 2012), and the application of SHCC for bridge and building members has been implemented successfully in civil infrastructures. SHCC however need higher contents of cement than normal concrete in order to develop the interfacial bond characteristics and to account for the absence of coarse aggregates in the mixture design (Choi *et al.*, 2012). Using high cement amounts leads to increased heat of hydration, higher shrinkage and is more energy intensive (Altwair *et al.*, 2012). In addition, use of a high ordinary Portland cement (OPC) content has negative environmental impacts associated with the cement manufacturing process (Gartner, 2004; Turner and Collins, 2013). Partial replacement of OPC in SHCC by waste materials such as fly ash (FA) has been reported in some studies as a possible solution to this problem (Nematollahi et al., 2015).

Lee Bang Yeon *et al.*, (2012) examined the feasibility of creating an engineering cementitious composite, considering environmental sustainability considerations, by using high FA content (up to 85% by weight) cement. Their results showed that a high

volume of FA can reduce the drying shrinkage, crack width, and improve tensile ductility, although this also reduced the 28 days compressive strength. Choi *et al.*, (2012) investigated the effect of partial replacement of cement by recycled materials on the mechanical properties of strain hardening cementitious material (SHCC). Alternative by-product materials (FA, sand, and polyethylene terephthalate (PET) fibres) were used to partially replace cement, silica sand, and polyvinyl alcohol (PVA) fibres, respectively. The results from this study indicated that FA improves both bending and tensile behaviour due to generation of stronger bonding between the PVA fibres and cement matrix. The SHCC containing PET fibres however performed relatively poorly in tensile and bending strength tests.

Recently, a feasibility study was carried out to develop a geopolymer-based engineering cementitious composite, known as engineered geopolymer composite (EGC), where the OPC binder was fully replaced by a slag-based geopolymer binder, which showed strain hardening and deflection hardening behaviors (in bending and uniaxial tension, respectively) accompanied by multiple cracking (Lee Bang Yeon *et al.*, 2012). Ohno and Li, (2014) was conducted to develop engineering geopolymer composite by replacing Portland cement material with fly ash. The results showed strain hardening and multiple cracking behaviour. However, the developed fly ashbased EGC possessed low to moderate compressive and uniaxial tensile strengths, ranging from 17.4 to 27.6 MPa and 2.9 to 3.4 MPa, respectively, which may limit widespread application of these composites in the construction industry.

2.7.3 Durability properties of fibre reinforced concrete.

Most previous research has focused on the mechanical properties of FRGC, and much less research has been conducted the durability performance of this material. For successful structural application durability requirements also need to be satisfied together with mechanical properties (Shaikh, 2013b). Yunsheng *et al.*, (2008) studied the freeze-thaw and acid resistance durability of steel fibre reinforced concrete with fly ash. For the freeze-thaw test, SFRGC specimens were subjected to 20 freeze–thaw cycles followed by an impact test. Based on the test results and analysis, the steel fibre FRGC exhibited excellent resistance to freeze–thaw cycles. These authors also reported some experimental results in terms of impact behaviour of SFRGC extruded panels after subjection to sulphuric acid solution for about a month. Their results showed that after subjection to acid attack the impact strength and stiffness of the composite without fly ash was reduced by 4.5% and 11.8%. The impact resistance of SFRGC with various percentage of fly ash, particularly for composites containing relatively high fly ash , 30% - 50%, was not reduced after acid attack (Shaikh, 2013a).

Puertas *et al.*, (2003) examined the shrinkage performance and residual strength after freezing/thawing and wet/dry cycles of PP fibre reinforced FRGC composite. Among different source materials the fly ash based FRGC exhibited lowest shrinkage at all ages up to about 40 days under both humid (RH > 95%) and dry (RH = 50% in laboratory) conditions. Under controlled laboratory dry conditions both slag and slag/fly ash based FRGC exhibited similar shrinkage to that of cement based FRCC. The effect of cyclic wet/dry cycles on the impact behaviour of FRGC was also evaluated by the same authors. Their results showed that, similar to shrinkage results, the fly ash based FRGC exhibited a 200% increase in the number of impacts required to create the first crack after 50 wet/dry cycles. The slag based FRGC material exhibited only a 50% increase in the number of impacts required to create the first crack after 50 wet/dry cycles.

Karahan and Atiş, (2011) studied the drying shrinkage and freeze-thaw resistance of concrete containing fly ash and polypropylene (PP) fibres. The fibre volume fraction was 0%, 0.05%, 0.10% and 0.20% on a volume basis, and the fly ash amount used in the concrete mixture was 0%, 15% and 30% by weight. Results showed a positive interaction between fly ash and polypropylene fibres resulting in the lowest drying shrinkage of fibrous concrete with fly ash. Freeze-thaw resistance of PP fibres concrete was found to slightly increase compared to concrete without fibres. In addition, fly ash addition increased the freeze-thaw resistance more than PP fibres addition.

Ganesan *et al.*, (2015) investigated the durability characteristics of plain and steel fibre reinforced geopolymer concrete and compared this with OPC conventional concrete. Fly ash was used the source material and a mixture of sodium silicate and sodium hydroxide solution was chosen as the alkaline liquid for producing the geopolymer. The covered specimens were given a rest period of one day and then placed in an oven

and cured at 60 °C for 24 hrs. The durability parameters considered in this study include water absorption, abrasion resistance, resistance to chemical attack, effect of alternate wetting and drying and resistance against chloride ions. Test results revealed that plain and fibre reinforced geopolymer concrete possesses superior durability characteristics compared to conventional concrete of the same grade with respect to most of the durability parameters.

2.8 Strengthening of concrete structures.

In recent years, strengthening of existing building and infrastructures that have failed prematurely due to rapid deterioration is making up an increasing portion of construction activities. Reinforced concrete structures show poor performance in terms of structural behaviour and durability under severe environmental conditions and high mechanical loading (Martinola *et al.*, 2010; Mourad and Shannag, 2012). This includes damage to bridges, buildings, parking structures, environmental facilities, as well as other structures. Therefore, the strengthening of deteriorated concrete structures is a major problem from a sustainability point of view to extend service life, otherwise the structure has to be demolished and reconstructed (Safdar *et al.*, 2016). Several methods and materials have been used for the repair and strengthening of reinforced concrete structures that are yielding excellent results for some specific applications, but still there is a need to develop a material capable of extending the service life in severe environmental conditions with a minimum of maintenance.

2.8.1 Typical reinforced concrete deterioration.

There are a number of causes of concrete structure failure:

- **Design or construction related problems**. Use of poor quality concrete, Insufficient joints, or construction defects are typical examples that fall into this group.
- Corrosion-related deterioration. Corrosion of embedded reinforcing steel is the most common cause of concrete deterioration. When the iron in steel bar is exposed to oxygen, water, and chlorides, it oxidizes and produces corrosion (rust). The corrosion of the steel reinforcement and expanding corrosion products results

in intense forces in the surrounding concrete, leading to additional cracking and/or delamination that accelerates the corrosion process.

- Environmentally-related problems. Structures located along seacoasts, or in northern climates where de-icing salts are used, can suffer accelerated deterioration. The most common causes of deterioration that form part of this group are alkali-aggregate reactions, sulfate attack, carbonation and freezing-thawing cycles.
- Extraordinary actions: Deterioration caused by earthquakes, impacts or fire.
- **Structural loads.** Fatigue caused due to overloading by heavy vehicles or change in usage of the structure with higher load requirements.

2.8.2 Compatibility between concrete substrate and repair / strengthening material.

The most important factor that causes failure in repairs is incompatibility between the substrate concrete structure and the new concrete (Paul and SE, 2002; Muñoz, 2012). The main parameters of the repair and strengthening material to take into consideration when selecting materials for retrofitting of concrete structure are discussed below:

- a) Bond strength at interface. The bond strength between the existing structure and the new materials is vital for the success of the strengthening and repair technique. Some strengthening materials required use of adhesives, for instance slurries or epoxy resins, to make sure that a satisfactory bond with the substrate is achieved. The interface has to sustain the stresses that could be caused by different loading scenarios or restrained volume changes (Muñoz, 2012).
- b) Differential Shrinkage. Tensile stresses are developed in the existing concrete substrate because of the shrinkage restrain in the overlay material. These stresses could lead to cracks or delamination at the interface between the concrete substrate and overlay materials (Rangaraju and Pattnaik, 2008).
- c) Curing requirement/ setting properties. The repair and strengthening materials should have rapid setting and hardening in order to accelerate the repair construction procedure and reduce the cost.

- **d)** Mechanical properties. The overlay material has to show sufficient or high mechanical properties to carry and transfer the loads (Paul and SE, 2002).
- e) **Durability properties.** The final service life of the overlay materials and the overall success of it significantly depends on the performance of the material as a barrier against all the varying processes that could deteriorate the structure such as water or chemical penetration, abrasion and freeze-thaw deterioration (Emmons and Vaysburd, 1996).
- f) Constructability. It is desirable to use overlay materials with similar placing and curing treatment practices to the substrate concrete to reduce the potential failures resulting from mistakes in the phase of construction. For instant, the self-consolidating behaviour of UHPC provides an advantageous feature when considering placing without the need for vibration, and has good adhesion to the substrate without using any bonding agents (Denarié *et al.*, 2005). Fewer steps in the construction procedure could be correlated with a reduction in potential mistakes.
- **g) Cost.** The overlay material cost has a considerable impact on the final choice of the material utilized for repairing and strengthening concrete structures. However, it should not be taken into consideration over the performance characteristics. A poor choice of repair material would cause the earlier failure of the repaired member. Cost needs to be tied to the expected service life in order to have an adequate economic analysis of the repair (Muñoz, 2012).

2.8.3 Strengthening of concrete structures using fibre reinforced concrete.

The development of new, cost effective repair methods is crucial to prolong the service life of reinforced concrete structures. Several researchers have used fibre reinforced cementitious composites as a promising repair material. Many types of fibre reinforced cementitious materials such as fibre reinforced concrete (FRC), high-performance concrete (HPC) and ultra-high performance fibre reinforced concrete (UHPFRC) have been utilized to meet the requirements of sustainable infrastructures (Matsumoto *et al.*, 2002). Increasing requirements of load bearing capacity, durability, and safety concern of concrete structures also demand use of UHPFRC for repair and maintenance (Maca *et al.*, 2013). Fibre reinforced concrete has good flexural and tensile properties and ease of application, it may also be used to improve the ductility

and load carrying capacities of flexural members by strengthening them (Iqbal *et al.*, 2016).

Farhat *et al.*, (2007) investigated the effect of using UHPFRC strips for strengthening beams. Epoxy adhesive was utilized for bonding between substrate concrete and UHPFRC. Their results indicated that using UHPFRC increased the optimum load carrying capacity by 86% and prevented shear failure of the strengthened beams. Brühwiler and Denarie, (2008) and Brühwiler, (2012) reported the efficiency of using UHPFRC material for the strengthening of crash barrier walls of highway bridges, bridge piers, and industrial floors. Habel *et al.*, (2006) reported the efficiency of combination of UHPFRC with reinforcing steel bars for the strengthening of existing concrete elements as their carrying capacity and their ultimate moment were considerably increased (Lampropoulos *et al.*, 2016).

Mechtcherine, (2013) utilized 30 mm and 40 mm high performance fibre reinforced cementitious composites for repair of pre-cracked reinforced concrete beams and demonstrated the crack bridging effects of these layers. Esmaeeli *et al.*, (2013) utilized strain hardening cementitious composite for strengthening of flexural brick masonry members in aging structures and reported considerable enhancements in the peak load and ductility, with formation of multiple cracks. Cho *et al.*, (2015) mentioned considerable developments in flexural response, increase in the ultimate load carrying capacity and post cracking stiffness of reinforced concrete slabs cast with a combination of 20 mm and 40 mm strain hardening concrete composite (SHCC) layer in the tension zone along with normal reinforced concrete composite on top, compared to the respective flexural strength value of slabs made entirely of reinforced concrete composites.

The application of high performance concrete for bridge and building elements has been implemented successfully in civil infrastructures as mentioned earlier. However, compared to conventional concrete mixtures, FRC mixtures are energy intensive. In addition, high cement content in the mixture design usually creates negative environmental impacts due to the associated carbon dioxide emissions. Free cement (geopolymer) based fibre reinforced concrete could be a solution for better sustainability and resource use as well as for reducing energy demand. As noted earlier geopolymer mortar cured under elevated temperature has comparable strength properties to cement based concrete and superior durability characteristics. However, a number of performance characteristics such as fibre reinforced concrete cured under room temperature have not been examined and need to be addressed. Moreover, the structural properties of fibre reinforced geopolymer in term of carrying capacity, bond and compatibility have not been examined.

2.9 Summary and conclusions.

The review of the literature was undertaken to show the state-of-the-art of the current understanding of geopolymer concrete (and in particular why researchers have looked for alternative binders to conventional Portland cement), and what are the challenges facing use of geopolymer concrete such as curing conditions, strength development and durability. The examination of the literature reveals the following key observations:

- Portland cement is one of the major cementing materials used to produce conventional concrete. However, there are number of disadvantages associated with the process of cement manufacturing such as raw material and energy consumption, and emission of significant amounts of CO₂. As the world moves towards sustainable development governments are starting to develop low carbon polices by applying carbon taxes on cement industries. Therefore, it is important to find alternative green energy binders such as geopolymer material to ordinary Portland cement.
- Fly ash is an industrial waste material from coal power plants, and slag is a byproduct from steel and iron mills. These materials are commonly available and could be used in producing geopolymer concrete. Silica fume (SF) is a by-product of the smelting process in the silicon and ferrosilicon industry. Silica fume is commercially available in a range of particle sizes which show varying behaviour effects on the performance of concrete.
- Geopolymers, a new form of binder used in cement and concrete composites, are produced by the reaction of aluminosilicate material with alkaline solutions. Fly ash and slag have become common source materials for geopolymers due to high

silica and alumina contents and are abundantly available in landfill sites. Potassium silicate and sodium silicate are usually used as alkaline activators for geopolymer binder. Geopolymer concrete has many environmental benefits including reduced CO₂ emissions and energy consumption compared with Portland cement concrete.

- Fly ash based geopolymer concrete has comparable mechanical properties to Portland cement under elevated temperature curing, although the geopolymerization process has low reaction rates under ambient temperatures.
- Several previous studies reported the superior durability performance of geopolymer materials in term of chemical resistance, chloride penetration and fire resistance. However, to date most of the published work focuses on geopolymer concrete cured under elevated temperatures, which again limits the application of this material to precast elements. There is limited study address the effect of curing temperature on the durability characteristics of geopolymer materials.
- Research has indicated that addition of discontinuous fibres to Portland cement concrete provide improved performance due to their high elastic modulus and ability to form a strong bond with the surrounding cementitious matrix. Use of fibre reinforced concrete is an efficient method to develop the mechanical properties of quasi-brittle, cementitious materials such as ductility, durability, energy absorption, fatigue, and toughness. A review of the literature has shown that very few studies have been performed related to fibre reinforced geopolymer composites cured under room temperature.
- Previous research indicates that strengthening existing buildings and other infrastructure using fibre reinforced concrete is a promising technique, since existing structures were efficiently strengthened and their resistance and their ultimate moment were considerably increased.

The current state of the art shows that development of geopolymer material as an alternative cementitious material to Portland cement is key to reduce the environmental footprint of the construction sector, besides providing improved or comparable mechanical and durability properties to conventional concrete. There is a lack of broad reference data for geopolymer materials cured under room temperature and this thesis intends to help to fill this gap. Most previous studies have examined the

behaviour of geopolymer composites cured under elevated temperature, which is problematic when producing geopolymer at a large production scale, and for in-situ, cast in place applications. A number of authors have examine the development of geopolymer mixes with a variant combination of fly ash and slag in the mixture and in most of these studies promising results were achieved. However, in these studies high volumes and concentrations of corrosive alkaline activator have been used leading to geopolymer products with potential health and worker safety issues during application. This study investigates the development of more user friendly geopolymer mortar and fibre reinforced geopolymer composites by using ternary mixtures of geopolymer materials (fly ash, slag and silica fume), and the effect of variant particle sizes of silica fume to improved material microstructure and enhanced fibre-matrix interfacial properties. The effect of FRGC materials on structural behaviour has been rarely studied however and, to date, there are no published studies (to the author's knowledge) on the evaluation of the corrosion resistance and the efficiency of the use of reinforced FRGC layers for the strengthening of existing structural elements. This study examines the application of a newly developed fibre reinforced geopolymer concrete cured under ambient temperature for strengthening RC beams, and investigates the resulting improvements in load carrying capacity under standard and accelerated corrosion conditions. The interfacial bond strength, differential shrinkage, curing condition, mechanical loading and durability properties are examined to evaluate the potential use of FRGC for repair and strengthening of concrete structures. Cost and wider constructability, aspects have not been examined in this study, but could form the focus of future research (discussed in chapter 9).

CHAPTER 3:

METHODOLOGY, MATERIALS AND TESTING PROCEDURES

3.1 Introduction.

The details of the experimental programme are presented and explained in this chapter along with work packages for each chapter. Specifically, the characteristics and specifications of the materials, the mix proportions, and the mixing procedure, casting and curing of the test specimens are presented. The chapter also details the experimental methods used, where the sample types, the test program and the test parameters are explained.

3.2 Research methodology.

This study takes an experimental approach to develop and test plain geopolymer mortar and fibre reinforced geopolymer with improved structural characteristics, suitable for cast-in-place applications and for the structural strengthening of existing buildings. The research was structured into two stages; a material stage and a structural stage. The mechanical performance of fibre reinforced cementitious composite depends on both the fibres, and the cementitious matrix. Thus, in the first phase of the development of FRGC, the fresh, hardening and microstructural properties of user friendly geopolymer mortar was examined and then generation of an enhanced fibre matrix interfacial bond was assessed by examining different silica fume particle size and different slag contents. Finally, the strain hardening performance of the FRGC was evaluated by examining different fibre volume fractions, aspect ratios and fibre types. Results from this study provide insight into the feasibility of using FRGC as strengthening and repair material under ambient temperature.

In the second stage, the efficiency of a newly developed fibre reinforced geopolymer concrete for repair and strengthening RC beams was investigated, and the resulting improvements in load carrying capacity under standard and accelerated corrosion conditions were examined. The mechanical performance of reinforced concrete beams strengthened with FRGC was investigated using four-point bending tests. Large scale beams strengthened with additional FRGC layers reinforced with steel bars have been examined. Moreover, FRGC with two variant depths was overlaid on RC beam

substrates to simulate a repair surface coating, in order to investigate the effect of overlay depth on corrosion protection performance. Respective specimens strengthened with conventional RC layers have been examined in order to evaluate the effectiveness of (or improvement generated over conventional techniques by) the proposed technique. The experimental results include the failure mode, load-deflection response and interfacial bond. Steel mass loss due to corrosion processes is also examined. The experimental, rather than theoretical or modelling approach has been utilised in this research, following previous studies (Wallah and Rangan, 2006; Adam, 2009; Rickard, 2012a; Buss, 2013). This is referred to alternative approach due to the complexity of the ternary geopolymer binder material with different fibres, and the lack of standard codes for fibre reinforced geopolymer concrete or even geopolymer material generally, for material characterisation. This is further reflected on in chapter 9.

3.3 Outline of the experimental programme.

The experimental study consisted of four main phases:

3.3.1 Phase I: mix design of plain geopolymer mortar cured under ambient temperature.

The first phase of the experimental program aimed to improve plain geopolymer mortar cured under ambient temperature with sufficient mechanical properties for structural applications. Initially, the work identified the role of various parameters which may affect material compressive strength and fresh properties in order to enhance the overall performance. Effect of mixing procedures, curing time, curing temperature, water content, superplasticizer content and alkaline content on the workability, setting time and compressive strength of binary blended geopolymer mortar cured under ambient temperature were investigated.

The impact of binder compositions (different slag content and silica fume types) incorporation on the fresh properties (setting time and workability), hardening properties (porosity, compressive strength, tensile strength and flexural strength tests), physical and chemical properties (SEM, FTIR, thermal analysis) of binary and ternary blended geopolymer mortar cured under ambient temperature were investigated.

3.3.2 Phase II: development of FRGC with enhanced mechanical properties and strain hardening performance.

For structural applications, post cracking performance of fibre reinforced concrete is important. Achieving a high strength fibre reinforced geopolymer concrete with strain hardening characteristics cured under ambient temperature is quite challenging. The low strength properties of the geopolymer matrix leads to weaker interfacial bonding between fibre and geopolymer matrix. Therefore, in the first part of this phase of the research work, the bond characteristics between fibre additives and the geopolymer matrix were improved by modifying the mechanical strength of the geopolymer matrix. A ternary binder mixture of fly ash, slag and variant silica fume types (densified, undensified and slurry) was examined in order to optimise the mechanical performance of the geopolymer matrix. Following this, the effect of slag content and silica fume particle size on the interfacial bond properties of steel fibre reinforced geopolymer composites (SFRGC) was examined. In all the examined mixtures, a 2% volume fraction of steel fibre was used with 13 mm length and 0.16 mm diameter.

In the second part of this phase, five types of fibre in FRGC were examined, 13 mm straight steel fibre, 6 mm straight steel fibre, 50 mm hook end steel fibre, PVA fibre and glass fibre. These fibres perform in different ways - the PVA fibre has a hydrophilic surface, low modulus, and high strength, and has been claimed to form a good chemical bond with the matrix. Steel fibre is the most common fibre used in fibre reinforced concrete for structural applications. It has high tensile strength to increase the first peak strength, while hooked end fibres are used to develop mechanical interlock. The effect of these fibres at different volume fractions on FRGC performance were examined using compressive strength, tensile strength, and flexural (comprising flexural strength, fracture energy and flexural toughness) testing of FRGC. In addition, the microstructural interfaces were examined using scanning electronic microscopy (SEM). The results from these mixes were compared with the mechanical properties obtained from plain geopolymer mortar and Portland cement mortar.

3.3.3 Phase: III durability properties of FRGC materials.

The optimum volume fraction of each fibre type of fibre reinforced geopolymer concrete mixtures (in term of mechanical performance) were further investigated regarding their durability characteristics in severe exposed environments. In the examined mixes, varying types of fibre were examined and compared with Portland cement based conventional mortar and plain geopolymer mortar. Four experiments were conducted: shrinkage (free shrinkage, end restrained shrinkage and overlay shrinkage), corrosion resistance, rapid chloride migration test and chemical resistance (sulphuric acid and sodium sulphate).

3.3.4 Phase IV: investigation of structural behaviour following repair and strengthening using FRGC.

The final phase of the experimental program focused on the structural application of fibre reinforced geopolymer concretes at larger scales. Fibre reinforced geopolymer concretes were used as strengthening and repair materials for the protection of the steel bars of a new layer, and for the subsequent improvement of the flexural strength of existing reinforced concrete beams. Large scale beams strengthened with additional FRGC layers reinforced with steel bars were examined. Also, an additional investigation has been conducted in beams where part of the concrete cover at various depths has been replaced by FRGC. In all the examined cases, respective beams with conventional concrete have been examined in order to evaluate the efficiency of the proposed technique. Accelerated corrosion tests were performed using an induced current technique by applying a nominal constant anodic current.

3.4 Materials.

Fly ash was used in this study as the main binder, and ground granulated blast furnace slag (GGBFS) and silica fume (SF) were used as a partial replacement in production of the fly ash based geopolymer materials. A mixture of potassium hydroxide solution and potassium silicate solution was used as the alkaline activator. Silica sand with particle size less than 0.5 mm was used as fine aggregate. The following sections discuss in detail the material properties of each mix ingredient.

3.4.1 Fly Ash.

Fly ash conforming to BS EN 450-1, (2012) (fineness category S and loss on ignition category B) or equivalent to low calcium class F in ASTM standard C618) was used as an aluminosilicate source in this study. Fly ash (FA) was supplied from the Drax Power Station, North Yorkshire, UK under the Cemex brand (Figure 3.1). The chemical compositions of the fly ash are presented in Table 3.1. The sum of silicate SiO₂ and aluminate Al₂O₃ is greater than 70 percent of total composition.

Chemical compositions	Fly ash	Slag	OPC	Silica	Silica
(%)				Sand	fume
Silicon Dioxide, SiO ₂	59	35	12.22	99.73	>90
Aluminium Oxide, Al ₂ O ₃	23	12	3.85	0.1	
Calcium Oxide, CaO	2.38	40	73.82		
Ferric Oxide, Fe ₂ O ₃	8.8	0.2	2.85	0.051	
Sulphur Trioxide, SO ₃	0.27		5.3		
Sodium Oxide, Na ₂ O	0.74			< 0.05	
Potassium Oxide, K ₂ O	2.81		1.17	0.01	
Magnesium Oxide, MgO	1.39	10	0.78		
Loss on ignition, LOI	6.7			0.09	<3

Table 3.1: Chemical compositions of FA, GGBS and Silica Sand.



Figure 3.1: Fly ash powder.

3.4.2 Ground Granulated Blast Slag.

GGBS was used to partially replace fly ash geopolymer binder. GGBS is used as a source of calcium and aluminosilicate in the geopolymeric binding system. The chemical compositions of the slag received from the manufacturer are presented in Table 3.1. The GGBS used in this study was supplied by Hanson UK, as a light coloured near white powder (Figure 3.2).



Figure 3.2: GGBS powder.

3.4.3 Silica Fume Types.

Various types of commercial silica fume (SF) with different physical properties were utilised in this study. Silica fume as received from the manufacturer Elkem material, UK is presented in Figure 3.3, and as supplied chemical composition is given in Table 3.1.



Figure 3.3: Silica fume types; densified silica fume (a), slurry silica fume (b) and undensified silica fume (c).

3.4.4 Ordinary Portland cement.

Commercially available CEM II Portland cement (from Hanson UK) compatible with EN 197-1 was used as a binder to make the control Portland cement mixture which was used for comparison (Figure 3.4). The chemical compositions of the ordinary Portland cement are presented in Table 3.1.



Figure 3.4: OPC powder.

3.4.5 Silica sand.

Fine washed, graded and dried silica sand was used in this study as a fine aggregate, and was obtained from Sibelco UK (Figure 3.5). The particle size distribution of the < 0.5 mm fraction is shown in Figure 3.6. The chemical composition is presented in Table 3.1.



Figure 3.5: Fine silica sand.



Figure 3.6: Particle size distribution of fine Sand.

3.4.6 Water and Admixtures.

Potable tap water in accordance with BS EN 1008:2002 was used to cast all geopolymer mortar and fibre reinforced geopolymer concretes. After different types of high range water-reducing admixtures were examined, the required workability was achieved by using polycarboxylate-based superplasticizer. The Viscoflow 2000 brand (provided by Sika UK (Figure 3.7) and meeting the requirements of BS EN 934-2, (2009) was used.



Figure 3.7: Sika viscoflow 2000 superplasticizer.

3.4.7 Alkaline Activator.

A mixture of potassium hydroxide pearl (85% purity) and commercially available potassium silicate solution was used as an alkali activator. A potassium based solution was chosen rather than the more common sodium-based solutions because potassium-containing alkaline activator showed double the compressive strength of a metakaolinite-geopolymer with the same quantity of sodium alkaline solution (Davidovits, 2011).

3.3.7.1 Potassium Hydroxide Pellets.

A 8M KOH solution has been considered to provide the required molarity of the final alkaline activator composition. Pellets of 85% purity obtained from Sigma Aldrich UK were used (Figure 3.8).



Figure 3.8: Potassium hydroxide container (left) and pellets (right).

3.3.7.2 Potassium Silicate Solution.

A commercially available potassium silicate solution (Figure 3.9) was obtained from Tennants Distribution UK. The composition of the solution is: modulus ratio $SiO_2/K_2O = 2.23$, water content= 45-65 wt. %, specific gravity 1.6 g/mL (Figure 3.9).



Figure 3.9: Potassium silicate solution.

3.3.7.3 Preparation of Alkaline activator.

The preparation of alkaline activator chemicals involved two alkaline products, potassium hydroxide solution and Potassium silicate solution. The Potassium hydroxide solution was prepared by dilution of potassium hydroxide pellets with distilled water in a fume cupboard (Figure 3.10a). Potassium hydroxide is classified as a corrosive product which has the potential to seriously burn skin, eyes and internal organs, therefore special care has been taken during handling and working with the substance. The solution was left for 24 hours to cool down to room temperature before

mixing with potassium silicate solution (mass of potassium silicate solution / Potassium hydroxide solution=2.5), to form a solution modulus (Ms=SiO₂/K₂O) of 1.25. The produced potassium silicate was left in the fume cupboard for 24 hrs before use (Figure 3.10b).



Figure 3.10: Alkaline activator preparation; dissolving Potassium hydroxide (a) and mixing Potassium silicate solution with Potassium hydroxide solution (b).

3.4.8 Fibres.

Five types of fibres were examined in this study: two different high strength brasscoated steel fibres (6 mm and 13 mm lengths), hooked steel fibre, Polyvinyl Alcohol (PVA) fibre and glass fibre (Figure 3.11). Their properties cover a wide range of mechanical properties, as illustrated in Table 3.2. The PVA fibres were supplied by Kuraray of Japan while steel fibre was supplied by Bekaert Dramix, UK.



Figure 3.11: Fibres used in the current research.

Fibre type	Geometry	Length	Diameter	Aspect	Fibre	Density	Е
		[mm]	[mm]	ratio	strength	$[Kg/m^3]$	[GPa]
				[L/D]	[MPa]		
Steel (ST6)	Micro	6	0.16	37.5	2250	7850	200
Steel (ST13)	Micro	13	0.16	81.25	2250	7850	200
Steel (HE)	Macro	50	1	50	1150	7850	200
Glass	Micro	13	0.13	100	1620	2700	74
PVA	Micro	12	0.015	800	1560±325	1300	29.5

Table 3.2: Fibre properties.

Preparation and preconditioning. 3.5

3.5.1 Mixing procedures.

For the trial mixing of geopolymer mortars a 5 L Hobart mixer was used, while for the mixing of the examined specimens a Zyklos 75 L mixer was used (Pan Mixer ZZ 75 HE). The mixing procedures of the geopolymer mortar were slightly different with the different silica fume forms utilized and either with the presence of fibre or not. Thus, the mixing procedures in detail are presented with the relevant testing programmes in Chapter 4 and 5. In general, the alkali activator potassium silicate solution was prepared in advance and mixed with water and superplasticizer 5 min before addition to the solid materials (binder). Finally, sand was added to the mixer (Figure 3.12).



4

Mixing the solid powder binder together



Prepared Potassium silicate with MR 1.25 24hrs in advance before mixing with binder



Mixing the solid binder with



Finally added Silica Sand



Geopolymer cubic

Alkaline activator by Hobart Mixer



3.5.2 Casting and Curing.

The fresh geopolymer mortar was then cast into cubic, dogbone and rectangular beam specimens. The geopolymer mortar was placed in the moulds in two layers and compacted on a vibration table for 30 seconds.

After casting, all the moulds were sealed with plastic sheet to prevent excessive loss of moisture and were left in an ambient environment for a period of 24 hours to allow the geopolymer mortar to be fully set, after which they were demoulded. The geopolymer mortar specimens were then cured under room temperature up to the testing date.

3.6 Experimental methods.

The details of the experimental test program are presented and explained, along with each thesis phase, as follows

Work of phases I and II (Chapters 4, 5 and 6);

Examine the fresh, mechanical and microstructural properties of plain geopolymer mortar and fibre reinforced geopolymer concrete cured under ambient temperature

- Examine the fresh properties of the plain geopolymer mortar through assessment of workability and setting time.
- Determine the mechanical properties using compressive strength, tensile strength, porosity and flexural strength tests.
- Determine the physical characteristics and chemical compositions of all primary materials i.e. fly ash, slag and silica fume, and plain geopolymer mixture by particle size distribution analysis, SEM-EDS, thermal analysis and FTIR techniques.

Work of phase III (Chapter 7);

• Examine the durability properties of fibre reinforced geopolymer concrete (FRGC) using variant types of fibre reinforcement (straight steel, PVA, and Glass) cured under ambient temperature.

Work of phase IV (Chapter 8);

• Structural Properties of reinforced concrete beams under four-point flexural loading.

3.6.1 Work of phases I and II (Chapters 4, 5 and 6).

3.6.1.1 Fresh properties.

For each mortar mixture, two different tests were used to assess the fresh state mortar: setting time and workability.

3.6.1.1.1 Setting time.

The setting times (initial and final) of the fresh geopolymer mortar were evaluated using a Vicat needle as described in BS EN 480-2, (2006). The Vicat was set up with the needle shown in Figure 3.13, attached and calibrated by lowering to rest on the base plate and adjusting the pointer to the zero reference of the scale.



Figure 3.13: Setting time apparatus.

The geopolymer mortar was placed in the mould immediately after mixing and the penetration distances were measured every 5 minutes due to rapid setting of the examined mixtures. The time measured from completion of mixing until the time at which the distance between the needle and the base plate is 4 mm is the initial setting time. The final setting time was determined from completion of mixing until the time after which the needle no longer penetrates 2.5 mm into the specimen (BS EN 480-2, 2006). The determined values were calculated as the average of two separate tests under room temperature curing conditions (21–23 °C) (Al-Majidi *et al.*, 2016b).

3.6.1.1.2 Flowability test.

Flowability tests were conducted according to BS-EN-1015-3, (2007) and ASTM C 1437-07, (2008). Flow tests were conducted immediately after mixing. A conical brass mould was placed at the centre of the testing table and filled with geopolymer mortar in two layers. Each layer was tamped 20 times with a tamping rod to ensure uniform filling of the mould. When the mould is removed, the mortar changes from a conical shape with a 100mm base to a "pancake" shape. The mortar is vibrated as the flow table rises and drops, through a height of 12.5 mm, 25 times in 15 seconds. The geopolymer flow is the resulting increase in average base diameter of the mortar mass, measured on at least four diameters at approximately equally spaced intervals (Figure 3.14) (Kondraivendhan and Bhattacharjee, 2015).



Figure 3.14: Flowability apparatus.

3.6.1.2 Testing of Mechanical Properties.

Four different tests were conducted to measure the strength characteristics of the hardened plain and steel fibre reinforced geopolymer: compression tests, flexural strength tests, direct tensile tests and porosity tests.

3.6.1.2.1 Compressive strength test.

Compressive strength tests were conducted according to ASTM C109/109M, (2007), using a compression machine (Avery Denison 7227) with a capacity of 2000 KN and a constant loading rate of 45 KN per minute. Three cubic specimens with 50mm sides were used to determine compressive strength for each curing age.



Figure 3.15: Cubic compressive testing.

3.6.1.2.2 Tensile strength test.

a) Plain geopolymer mortar.

Geopolymer tensile performance was evaluated through direct tensile tests according to AASHTO T132, (2000). The examined dog bone-shaped briquettes had a thickness of 25 mm, a length of 76 mm and a mid-length cross sectional area of 625 mm² (Figure 3.16a). Special self-aligning grips were used for the direct tensile tests to ensure uniform stress distribution. The extension of the specimens during the testing was monitored using a Linear Variable Differential Transformers (LVDTs) attached to the specimens in order to exclude any induced slip at the grips (Figure 3.16b). The direct tensile tests were performed under a displacement rate of 0.4 mm per minute.



Figure 3.16: Direct tensile test of plain geopolymer mortar; specimen schematic (a) and test set up (b).

b) Fibre reinforced geopolymer concrete (FRGC).

Direct tensile strength was determined using 'dog bone' shaped samples of 13 mm (mid cross section) by 50 mm (Figure 3.17). The samples were manufactured to fit into a pair of clamps that were attached on both sides of the sample to measure displacement alongside the narrow cross section. The test was carried out to determine the tensile strength of geopolymer specimens reinforced with micro fibres after 3, 7, 14, 28 and 90 days curing. The setup shown in Figure 3.18 was used to determine the average displacement over a gauge length of 105 mm. The average extension was measured using Linear Variable Displacement Transducers (LVDT) attached to a steel frame, as shown in Figure 3.18. An Instron testing machine was used for the direct tensile tests and the tests were performed under displacement control with a rate of 0.4 mm/min which is in agreement with the loading rate used by (Hassan *et al.*, 2012) leading to comparable results. Measurements were recorded until the ultimate load was achieved. The load carrying capacity behavior after cracking of the fibre reinforced sample was also examined (Hassan *et al.*, 2012).



Figure 3.17: Dog bone shape specimen of FRGC.



Figure 3.18: Direct tensile set up of FRGC; front view (a) and side view (b).

3.6.1.2.3 Flexural strength test.

a) Plain geopolymer mortar.

Flexural strength tests were performed according to ASTM C293-02, (2002) using a simple beam with centre loading. For each mixture, Prismatic specimens with 75 x 75 x 285 mm³ dimensions were prepared (Figure 3.19a) and three specimens of each mixture were tested at curing ages of 7, 14, and 28 days to determine the average flexural strength. An Instron universal test machine was used and the setup is presented in Figure 3.19b.



Figure 3.19: Layout of the specimens employed for centre point bending test.

b) Fibre reinforced geopolymer concrete (FRGC).

Flexural strength was determined by testing standard prismatic specimens (100 x100 x 500mm) at 28 days using an Instron testing machine. A span length equal to 450 mm was used, with distance between the loading points set at 1/3rd of the span length (Lee *et al.* 2014). The flexural test set-up is shown in Figure 3.20. The testing machine was operated in a 'closed loop' mode to load the prisms at a fixed deflection rate of 0.24 mm/min. Two Linear Variable Displacement Transducers (LVDTs) were attached to a yoke frame which was used in order to eliminate any induced displacements at the supports during loading (see Figure 3.20). The load deflection curve was used to characterize the ultimate load and its related deflection, ultimate flexural strength, toughness and residual strengths based on ASTM C1609 / C1609M-05, (2005).



Figure 3.20: Bending specimen geometry.

3.6.1.2.4 Porosity testing.

Sample porosity was measured via the vacuum saturation technique, as reported in previous studies (Gonen and Yazicioglu, 2007; Keleştemur and Demirel, 2010). Porosity tests were carried out on at least two cylindrical samples (20 mm x 50 mm) for each mixture. The test procedure was as follows: prior to testing, the samples were dried in an oven at a temperature 105 ± 5 °C for approximately 24 hours, and then weighed. The samples were then kept in a vacuum, for 24 hours. The desiccator was then filled with distilled water so that samples were fully submerged in water. Then the samples were kept under vacuum for 24 hours and allowed to equilibrate for the next 24 hours (Figure 3.21). The specimens were then weighed in air and water (Khan,

2007). The amount of water penetrating into the sample is a measure of the porosity and is calculated as follows:

$$P = \frac{(W_{sat} - W_{dry})}{(W_{sat} - W_{wat})} x \ 100 \tag{3-1}$$

Where *P* is vacuum saturation porosity (%); W_{sat} is weight in air of saturated sample; W_{wat} is weight in water of saturated sample; and W_{dry} is weight of oven-dried sample.



Figure 3.21: Pressure saturation apparatus

3.6.1.3 Physical and microstructural analysis.

3.6.1.3.1 Particle Size analysis.

Particle size distribution analysis and specific surface area measurement of fly ash, silica fume, and slag were performed using a Malvern Laser scattering particle size distribution analyzer (Figure 3.22), which is able to measure particle sizes from 0.02 to 2000 μ m. The samples of fly ash, silica fume, slag were taken from the laboratory in dry powder form and tested in a closed-area at room temperature.



Figure 3.22: Malvern particle size analyser.

3.6.1.3.2 Scanning electronic microscopy (SEM).

For SEM analysis, plain geopolymer mortar and FRGC samples were taken from cracked samples after the end of the tensile tests. The fractured samples were sputtered with carbon and imaged using scanning electronic microscopy (SEM) (Zeiss; model LEO 1455VP) (Figure 3.23) with an accelerating voltage range of 1–30 kV.



Figure 3.23: Zeiss scanning electron microscope used in the present study.

3.6.1.3.3 Fourier transform infrared spectroscopy (FTIR) analysis.

Functional groups of materials were characterised using infrared spectroscopy. Molecular vibrations, which correspond to the fundamental vibrations of the functional groups, were examined using characteristic infrared absorption bands (Yip and Van Deventer, 2003; Lecomte *et al.*, 2006). Fourier transform infrared spectroscopy (FTIR) analysis were carried out using a Perkin Elmer System series 2000 (Figure 3.24) spectrophotometer
in a frequency range of 4000–400 cm⁻¹ to identify the functional groups of the raw materials (fly ash, silica fume and slag) and geopolymer paste mixture. FTIR analysis was carried out on fine powder generated by grinding the geopolymer paste sample to reduce its particle size.



Figure 3.24: Perkin Elmer Fourier Transform Infrared Spectrometry unit used in the current research.

3.6.1.3.4 Thermal analysis.

For thermal analysis, tests were conducted using a Mettler Toledo differential scanning calorimeter (Figure 3.25). During this test the binder percentage was kept constant as in the mixtures and total sample mass was around 20 g; mixing was performed manually; and sample loading time was around 20 min.



Figure 3.25: Differential Scanning Calorimetry (DSC) set-up.

3.6.2 Work of phase III (Chapter 7) [Durability testing program].

Work package 2 examined the influence of fibre type (straight steel, PVA, and Glass fibres) on the durability properties of fibre reinforced geopolymer concrete (FRGC) cured under ambient temperature. The durability testing program included shrinkage, corrosion resistance, chloride penetration and chemical resistance tests. These are described below, and further details are presented in Chapter 7.

3.6.2.1 Shrinkage characteristics.

The shrinkage behaviour of FRGC specimens were evaluated through free shrinkage, overlay shrinkage and end restrained shrinkage tests.

3.6.2.1.1 Free drying Shrinkage.

Drying shrinkage properties of FRGC were determined in accordance with (Sathia *et al.* 2008; ASTM C490/C490M 2011). A series of prisms with size 75 x 75 x 285 mm were utilised to determine the drying shrinkage of FRGC, and the average value of the three specimens was computed as the final result. After specimen demoulding and curing for 2 days, the initial length of the specimens were measured, and these were then kept at room temperature $(20\pm2 \ ^{\circ}C)$ and $60\pm5\%$ relative humidity. 10 curing periods were used, and the length variation of the specimen was measured by a digital indicator at the end of each period to calculate the dry shrinkage strain. The testing apparatus for dry shrinkage assessment is presented in Figure 3.26.



Figure 3.26: Free shrinkage apparatus.

3.6.2.1.2 Overlay Restrained Shrinkage.

The function of the overlay restrained shrinkage test is to evaluate the crack width and distribution of fibre reinforced geopolymer concrete overlay resulting from the differential shrinkage with the bonded concrete surface substrate. For each mixture, two beams with an area of 100 x 500 mm² and depth of 100 mm were cast and left to mature for 6 months prior to overlaying to ensure that the beams would dry sufficiently so that at least a major part of the shrinkage would take place prior to testing. An expected consequence was thus that the restraint would be higher due to a more pronounced differential shrinkage. The surface preparation, overlay casting and placing DEMEC disks procedures are described in detail in Chapter 7. The device used to measure the shrinkage of each specimen was a DEMEC gauge (pictured below), which included the measuring device, placement bar, and calibrating bar (Figure 3.27).



Figure 3.27: Demec Gauge, Reference Bar, and Calibration Bar.

The DEMEC disks are 6.3 mm diameter and flat in shape with a small indention in the middle to accommodate the placing of measuring points for the DEMEC gauge. The placement of disks and the measurement method of the prisms are presented in Figures 3.28 and Figure 3.29, respectively. Once the disks were placed, the initial length was recorded and then the specimens were kept at room temperature to measure strain shrinkage over specific periods. Shrinkage strain is the extension expressed in relation to the original gauge length and is calculated through the following equation:

extension =
$$0.8 x R x 0.002$$
 (3-2)

$$Strain = \frac{extension}{gauge length}$$
(3-3)

Where, *extension* is the increase in length from the original gauge length, and *R* is the reading measured from the DEMEC gauge.



Figure 3.28: Placing of Measurement Disks with Reference Bar



Figure 3.29: DEMEC Gauge Placement on Measurement Points (a) and Close-up View of DEMEC Gauge Measuring Points (b).

3.6.2.1.3 End restrained Shrinkage.

This test method was employed in Kovler, (1994) and developed by Carlswärd, (2006). In this study $60 \ge 40 \ge 400 \mod 67$ FRGC in addition to plain geopolymer and OPC mortar specimens were fastened between two end grips. In this study a "new" set-up was modified as shown in Figure 3.30, consisting of a rectangular hollow section (RHS of $120 \ge 80 \ge 8 \mod)$, giving a smooth and slippery surface to ensure that restraint would only develop at the ends of the specimen. L-shaped supports of steel ($40 \ge 40 \mod 64 \mod 75$) were secured to the flange by means of bolts at 500 mm. The restraining bolts, which had a very tight fit, were fastened prior to each test to ensure that restraint would only develop at the ends of the concrete specimen. The casting steps and measuring procedure are presented in detail in Chapter 7. The

DEMEC points and measuring gauge are similar to the gauge used in the overlay shrinkage tests (Figure 3.30).



Figure 3.30: End restrained shrinkage specimens

3.6.2.2 Corrosion resistance.

The corrosion resistance of FRGC materials was evaluated through examining crack damage, steel mass loss, and post-corrosion flexural loading capacity. The corrosion process is accelerated by impressing a constant current between the rebar (anode) and a steel sheet (cathode). Concrete prisms with dimensions of 75 mm by 75 mm cross section and 285 mm in length, and deformed steel bars of 12 mm diameter and 500 mm in length, were used in this study. The detailed testing procedures are described in Chapter 7.

3.6.2.3 Non-steady State Chloride Migration (RCMT).

The resistance of plain geopolymer and FRGC mixtures to chloride-ion penetration was evaluated using the rapid chloride migration test (RCMT), according to the Nordic test method (NT Build 492, 1999), at a curing age of 90 days. The RCMT test is based on actual measurement of chloride ion penetration depths under an applied electrical charge. The experimental set up and testing procedures are presented in detail in Chapter 7.

3.6.2.4 Chemical resistance [Sodium sulphate and sulfuric acid resistance].

The sulphate and acid resistance of FRGC materials were evaluated through their posttesting visual appearance, and by measuring the residual compressive strength and change in mass after 3 and 6 months of immersion in sulphate and sulphuric acid solutions. There is no specific standard for acid resistance. Sodium sulphate resistance was carried out following the method of (Wallah and Rangan, 2006).

3.6.3 Work of phase IV (Chapter 8).

Work package four, the structural behaviour of fibre reinforced geopolymer concretes at larger scale application under standard and accelerated corrosion conditions were examined. Initial beam's (prior to strengthening) cross sectional dimensions were 100 mm by 200 mm and the length was equal to 1400 mm. The reinforcement consisted of two steel bars with a diameter of 10 mm ($2\Phi 10$) with a characteristic yielding stress value of 530 MPa in the tensile side. Stirrups of 8 mm deformed bars diameter (Φ 8) were used in the shear span at an interval of 90 mm with a measured yield strength stress value of 350 MPa and spacing 90 mm (Figure 3.31). Wooden forms were used to cast the beam test specimens. A thin layer of grease was applied to the internal surfaces to ensure easy stripping and to prevent water absorption. The reinforcements were placed inside the forms, providing a minimum clear concrete cover of 20 mm. The conventional concrete was cast in the beam moulds in two layers, and each layer was compacted using an air needle concrete vibrator for about 5 second for each insertion to consolidate the concrete by removing the entrapped air to the surface and allowing concrete to flow into corners, around the reinforcement rebar and flush against the form face. After casting, the specimens were sprayed with water and covered with plastic sheets and were stored at room temperature until the day of testing. The preparation and casting of the reinforced concrete beams is shown in Figure 3.31.

Fibre reinforced geopolymer materials were used as strengthening and repair materials for the protection of steel bars in a new material layer, and for subsequent improvement of the flexural strength of existing beams. RC beams strengthened with 50 mm layer in the tensile side reinforced with steel bars and three side jacket have been examined. Also, the performance of Reinforced concrete beams where part of the concrete cover was replaced by FRGC was at various depths (12% and 25% of the total RC beam depth) was investigated.



Figure 3.31: Preparations and casting steps of Initial beam for the strengthening and repaired applications.

Accelerated corrosion tests were performed using the induced current technique by applying a nominal 300 μ A constant anodic current for approximately 30–90 days between the reinforcement bar (anode) and a copper mesh (cathode) at the bottom surface of the container connected to negative terminal of the DC power supply. RC beams were tested as simply supported beams under a four-point bending loading with an imposed deflection rate of 0.24 mm/min. The clear span of all the RC beams was kept constant at 1200 mm. The slip at the interface of the strengthened beams was measured during the bending tests by using six digital micrometers with an accuracy of 0.001 mm. Methods used for examining the structural properties of reinforced/repaired concrete beams, in terms of specimen preparation, reinforcement details, loading conditions and accelerated corrosion procedures, are presented in detail in Chapter 8.

CHAPTER 4:

DEVELOPMENT OF BINARY GEOPOLYMER MORTAR CURED UNDER AMBIENT TEMPERATURE

4.1 Introduction.

Geopolymer concrete technology involves production of more environmentally friendly waste material-based concrete, which could be a viable solution for conventional concrete replacement. Typical fly ash based geopolymer concrete however requires high temperature curing treatment in order to develop sufficient early strength properties (Barbhuiya *et al.*, 2009), which is considered a severe limitation for cast-in-place concrete applications. Previous research indicates that the chemical reactions and strength development of geopolymer concrete are affected by a number of parameters such as chemical activator composition and dosage, raw materials, mixing condition, water content and curing conditions (Duxson, 2007; Joseph and Mathew, 2012; Ryu *et al.*, 2013).

Fly ash and Ground Granulated Blast Furnace Slag (GGBS) are two types of calcium aluminosilicate solid waste materials commonly used in geopolymer concrete. The main reaction product of alkali-activated cements for slag is calcium silicate hydrate (C–S–H) while for fly ash it is amorphous hydrated alkali aluminosilicate (Chi and Huang, 2013). Alkali activated slag has high strength but issues related to rapid setting and insufficient workability along with high values of dry shrinkage have been reported. In contrast, fly ash based geopolymer mortars cured under ambient temperatures usually exhibit slower early strength development and setting time, which is likely to be due to a low content of Iron oxide, calcium hydroxide (Ca(OH)₂), and reactive silica. These mortars, however, showed identical compressive strength (62–66 MPa) after 24 h curing at 60–75 °C. High temperature curing for at least 6 h is typically recommended for fly ash based geopolymer concrete in order to accelerate the pozzolanic reaction and develop the mechanical properties (Bakharev, 2005b; Somna et al., 2011). van Jaarsveld et al., (2002) studied the effect of various curing temperatures on the compressive strength of fly ash geopolymer concrete, and based on this study, doubling of strength was achieved when the curing temperature was

increased to between 50 °C and 80 °C compared to the respective value at 30 °C. However, heat curing treatment leads to increased cost and practical issues, preventing in situ application of geopolymer concrete at large scale.

There are a few published studies on geopolymer mixes with a variant combination of FA and GGBS in the mixture (Chi and Huang, 2013; Lee and Lee, 2013; Deb *et al.*, 2014; Jang *et al.*, 2014; Nath and Sarker, 2014; Marjanović *et al.*, 2015) and in most of these studies promising results (in terms of strength) were achieved. However, in these studies high volumes and concentrations of corrosive sodium silicate and/or sodium hydroxide have been used, leading to geopolymer products with potential health and worker safety issues during application (Davidovits, 2011). Davidovits *et al.*, (2014) propose a user friendly geopolymeric method in order to improve the strength of geopolymer cement as well as reduce costs by avoiding thermal activation and promoting easier handling applications. To date, however, there are not any published studies on the mechanical performance of 'user friendly' geopolymer cement paste of these mixes have been conducted.

The work presented in this chapter addresses thesis objective 1. User-friendly fly ash/ slag /K-silicate/H₂O based geopolymer materials were used to investigate the development of geopolymer mortar cured under ambient temperatures through the analysis of mechanical properties and developed microstructures. The research presented in this chapter has examined the main parameters affecting the behaviour of geopolymer concrete cured under ambient temperature, and has derived the mixture proportioning of geopolymer mortar to optimise its mechanical performance. The chemical and physical characteristics of the examined materials were discussed in chapter three.

4.2 Experimental procedures.

An extensive experimental study has been conducted on fly ash and slag based user friendly geopolymer mortars cured under ambient temperature (20 - 23 °C) with mechanical properties comparable to fly ash -only based mixtures cured under elevated temperature. This study was carried out in two phases. In the first phase, a total of 13 mixtures of geopolymer mortar proportions were prepared, and are listed

in Table 4.1. The effect of mixing steps and time, curing temperature, water content, superplasticizer and alkaline activator to binder weight ratio on the fresh properties and compressive strength of fly ash and slag binder was examined, the latter being kept constant at (Fly Ash 1:1 Slag). Four different mixing steps and times were implemented in order to select the optimum mixing procedure in terms of workability and strength (Table 4.2). The effect of curing conditions (room temperature and heat curing) on the strength development over curing time was also examined. Three masses of water to binder ratios (0.23, 0.25 and 0.28), four percentages of polycarboxylate superplasticizers to binder (0, 0.005, 0.01 and 0.015) and four alkaline activator contents (0.08, 0.01, 0.12 and 0.14) were used. The second phase of current study examined the main parameters (binder compositions) affecting the behaviour of geopolymer mortar cured under room temperature. The chemical and physical characteristics of the examined materials were discussed in chapter three.

Mix	Fly	Slag	K ₂ SiO ₃	Water	superplasticizer	Mixing	Curing Type
No.	Ash		/binder	/binder	/binder	Procedure	
1	0.5	0.5	0.12	0.25	0.015	Α	Room temperature
2	0.5	0.5	0.12	0.25	0.015	В	Room temperature
3	0.5	0.5	0.12	0.25	0.015	С	Room temperature
4	0.5	0.5	0.12	0.25	0.015	D	Room temperature
5	0.5	0.5	0.12	0.25	0.015	Α	60 °C heat curing
6	0.5	0.5	0.12	0.23	0.015	Α	Room temperature
7	0.5	0.5	0.12	0.28	0.015	Α	Room temperature
8	0.5	0.5	0.12	0.25	0	Α	Room temperature
9	0.5	0.5	0.12	0.25	0.005	Α	Room temperature
10	0.5	0.5	0.12	0.25	0.01	Α	Room temperature
11	0.5	0.5	0.08	0.25	0.01	Α	Room temperature
12	0.5	0.5	0.1	0.25	0.01	Α	Room temperature
13	0.5	0.5	0.14	0.25	0.01	Α	Room temperature

Table 4.1: Mixture compositions of fly ash/slag based geopolymer mortar.

4.3 Phase I: Initial experimental work [Optimization of the mix proportion].

The main objectives of this stage of study were:

- I. To select the mixing steps and time (Table 4.2);
- II. To obtain the basic mixture proportioning of geopolymer mortar;
- III. To evaluate the parameters that affect the geopolymer performance; and
- IV. To examine the fresh and hardened state of geopolymer mortar.

Test	Mixing Steps and time	Mixing time
		[minutes]
	Dry mixing solid GP binder= 5 min	
Mix	Liquid (water-K ₂ SiO ₃ - superplasticizer) to dry binder= 5 min	13
А	Added Sand= 3 min	
	Dry Mixing solid GP binder (fly ash) with Sand= 5min	
Mix	Liquid (water- K_2SiO_3 -superplasticier) to dry binder= 5 min	13
В	Added Slag to solution= 3 min	
	Dry mixing (fly ash alone) with Sand= 5 min	
Mix	Added Liquid (water-K ₂ SiO ₃ -superplasticier) to GP binder= 10	20
С	min	
	Added slag to solution= 5 min	
	Dry mixing (fly ash alone) with Sand= 5 min	
Mix	Added solution (slag-K ₂ SiO ₃ -water-superplasticizer)= 5 min	13
D	Added Sand= 3 min	

Table 4.2: Details of mixing Procedures.



Figure 4.1: Flow diagram of mixing process A.

4.3.1 Effect of salient parameters on the fresh properties of binary blended geopolymer mortar cured under ambient temperature.

For each mixture (Table 4.1), two different tests were used for the fresh state; setting time and workability. Initially, the effect of mixing steps and mixing time, water content, alkaline activator and superplasticizer content on the setting time of geopolymer mixes was investigated. In this test, two measurements were taken for the initial and final setting time as proposed by BS EN 480-2, (2006). Initial setting time was measured from completion of mixing until the time at which the needle penetrated to a distance of 4 mm from the base of the plate. Final setting time was measured at the point at which the needle penetrated to a distance of 2.5 mm.

4.3.1.1 Effect of mixing steps on the fresh properties of geopolymer mortar.

The four different mixing procedures tested considerably effect the fresh properties of geopolymer mortar. In all mixing procedures, the alkaline solution (Potassium silicate and potassium hydroxide) was prepared in advance and left overnight before use. However, the timing of addition of slag powder to the mixture was changed with different mixing procedures, as slag is considered a highly pozzolanic material which could play major role on the workability properties. Mixing procedure A involved adding the liquid phase (alkaline solution and water) to the dry solid material, while mixing procedure B added slag at the end of mixing steps. Generally, the mixing procedures A and B are widely used to manufacture geopolymer due to their simple use. The mixing procedures proposed by GEOASH (Davidovits, 2011) by mixing slag solution to the solid material have also been examined (Mix D). For mixing procedure C, the mixing time was extended from 13 minutes to 20 minutes in order to examine the effect of curing time on the geopolymer performance. The effect of mixing procedures on the fresh state performance of the geopolymer mortar in terms of flowability and setting time are presented in Figure 4.2.



Figure 4.2: Effect of mixing procedures on the Flowability and setting time of geopolymer mortar (mix D cannot be measured due to high viscosity that mixture D did not flow in the flow table test).

As can be seen from Figure 4.2, the mixing procedures considerably effected the fresh properties of geopolymer mortar. In term of setting time, the mixing procedure had a clear impact on the drying characteristics of geopolymer mortar. The results show that

in mix A, the initial and final setting time of geopolymer were 34 and 45 minutes, respectively. Adding slag at the end of the mixing steps (mix B) reduced the initial and final setting time to 30 min and 40 min, respectively. On the other hand, increasing mixing time (i.e. making the total mixing time 20 minutes, mix C prolonged the initial and final setting time to 35 and 50 min, respectively. Mixing slag with the liquid phase to form slag solution before addition to the solid binder (fly ash and sand) (Mix D) severely reduced the workability. The geopolymer mortar obtained was extremely viscous, meaning that the moulds used for the tests were not easily filled at the high slag content utilized in the examined mixture.

The results also shown that the mixing steps and time effects on the Flowability of geopolymer mortar. Increasing mixing time slightly improved the workability of the geopolymer mortar. It can clearly be seen that mixing processes A and C had similar fresh characteristics, whereas slag were mixed for longer time. However, adding slag at the end of the mixing steps resulted in an increase in moisture loss and fast solidification. Based on these fresh properties and the compressive strength results described in detail in section 4.3.2 of variant geopolymer mortar mixture procedures, mix procedure A was selected as the optimum mixing procedure and used subsequently the examined mixtures.

4.3.1.2 Effect of water content on the fresh properties of geopolymer mortar.

The effect of free water content on the fresh properties of user friendly geopolymer mortar is presented in Figure. 4.3.



Figure 4.3: Effect of water content on Flowability and setting time of geopolymer mortar.

In order to examine the effect of water content on the fresh properties of geopolymer mortar cured under ambient temperature, three geopolymer mixes were designed with different water content (water to binder weight ratio) of 0.23, 0.25 and 0.28. Based on the results of Figure 4.3, water content considerably affects the setting time of slag and fly ash based geopolymer mortar. The initial and final setting times of the 0.23 w/b mix were 23 min and 32 min respectively, while those of the 0.25 w/b mix were 24 min and 45 min. By increasing water to binder weight ratio to 0.28, both initial and final setting times were increased to 40 min and 77 min, respectively. The results also show that increasing water content in the mixture considerably increased the flow of the geopolymer mortar. The flow was increased by 55% and 100% when the water to binder weight ratios were increased from 0.23 to 0.25 and to 0.28.

4.3.1.3 Effect of superplasticizer content on the fresh properties of geopolymer mortar.

High range water reducing admixtures known as superplasticizers (SPs) are commonly used to reduce the water content in OPC concrete while maintaining a constant workability, resulting in higher strength and durability of concrete (Nematollahi and Sanjayan, 2014b). There are several types of superplasticizer such as naphthalene, polycarboxylate, lignosulphonates and melamine-based solutions. Some studies have examined the effect of superplasticizer types and content on the fly ash or slag based geopolymers (Hanehara *et al.*, 2001; Palacios and Puertas, 2005). The effect of superplasticizer content on user friendly geopolymer mortar cured under ambient temperatures have not however been examined. The effect of polycarboxylate based superplasticizer contents on the Flowability and setting time of fly ash and slag based geopolymer mortar are shown in Figure 4.4. Four different geopolymer mixtures with different superplasticizer content (of 0%, 0.5%, 1% and 1.5%) were examined.



Figure 4.4: Effect of superplasticizer content on the Flowability and setting time of geopolymer mortar.

Figure 4.4 shows a comparison of fresh properties of geopolymer in terms of setting time and workability for variant superplasticizer to geopolymer weight ratio. Based on the results of Figure 4.4, the initial and final setting times of geopolymer mortar without superplasticizer were lower than those for geopolymer mortar containing superplasticizer. The initial and final setting times were extended as the superplasticizer content was increased. Specifically, initial and final setting times of geopolymer mortar without superplasticizer were 20 min and 27 min, respectively. These increased to 30 mins and 40 mins respectively when 1% of polycarboxylate superplasticizer was added in the mix. The setting time was further increased with increasing superplasticizer content to 1.5%. These findings agree with the previous study of (Jang *et al.*, 2014).

The Flowability of fly ash and slag based geopolymer mortar with different superplasticizer content are also presented in Figure 4.4. The Flowability of geopolymer containing superplasticizer was significantly increased compared with the control geopolymer mixture (without superplasticizer). The flow increased by 50% and 80% for geopolymer mortar containing 1% and 1.5% superplasticizer compared to the mixture without superplasticizer. These results illustrate the retarding effect of polycarboxylate-based superplasticizer on setting time of geopolymer with high superplasticizer content.

4.3.1.4 Effect of alkaline activator content on the fresh properties of geopolymer mortar.

Mixtures 10 to 13 (Table 4.1) were prepared to study the effect of alkaline activator content on the fresh properties of geopolymer mortar. Results are shown in Figure 4.5. The details of four different mixtures designed with different alkaline activator contents 8%, 10%, 12% and 14% are listed in Table 4.1. The molar concertation of potassium silicate solution was kept constant (MR equal to 1.25) for all the examined mixtures.



Figure 4.5: Effect of alkaline activator content on the Flowability and setting time of geopolymer mortar.

The results indicate that the content of alkali activator has significant effects on the Flowability and setting time of geopolymer mortar. The geopolymer mortar mixtures with 8% potassium silicate content showed poor workability, fast setting times and were hard to cast compared to the other mixtures with higher alkaline activator content. Increasing alkaline activator from 10% to 12% improved the Flowability from 160 mm to 180 mm, respectively. The initial and final setting time increased from 25 min and 35 min to 30 min and 45 min when increasing alkali activator content from 10% to 12%, respectively. However, increasing the alkali activator content beyond 12% up to 14% did not show significant effect on fresh properties.

- 4.3.2 Effect of salient parameters on the compressive strength development of geopolymer mortar cured under ambient temperature.
- 4.3.2.1 Effect of mixing procedure on the compressive strength development of geopolymer mortar.

The mean compressive strength was calculated from three specimens for each mixture. Previous studies have indicated that the mechanical properties of geopolymer paste dramatically change with different mixing procedures due to different rates of geopolymer gel formation (Kobera, 2011). The effect of mixing procedures on the compressive strength development of user friendly geopolymer mortar cured under ambient temperature is presented in Figure 4.6.



Figure 4.6: Effect of mixing procedures on the compressive strength of geopolymer mortar.

As can be seen from Figure 4.6, mixing steps and time show considerable impact on the compressive strength of geopolymer mortar. At the same mixing time, dry mixing geopolymer binder (slag with fly ash) then adding the liquid phase to the solid phase (mix A) gives higher compressive strength than adding slag at the end of the mixing steps (mix B). However, for the same mixing procedure, increasing mixing time from 13 mins to 20 mins slightly improves the compressive strength of geopolymer mortar. The compressive strength of mixing procedure B was lower than that of mixing procedure and for a short time leads to rapid loss of moisture from the particle surface and an incomplete geopolymerization process. Increasing the mixing time of slag in the mixture provided a better uniformity and improved the fresh properties (Figure

4.2). The mixing procedure A (Table. 4.2) generates samples with the highest compressive strength, at around 19 MPa and 45 MPa at 3 days and 28 days, respectively. Mixing procedure A (Figure 4.1) is also recommended by European research GEOASH, 2008 (Davidovits *et al.*, 2014), as from the technical point of view mixing the solid binder together then adding liquid solution to the solid components is simple, and avoids complications in its operation at large scale.

The effect of curing time on strength development are also presented in Figure 4.6. The compressive strength values indicate a similar trend in the strength development under different curing times. At early ages (3 days and 7 days), the compressive strength of all examined mixtures was low, and there were significant differences between the three different procedures. After 14 days, the strength values increased and there was little difference between the three mixes.

4.3.2.2 Effect of water content on the compressive strength of geopolymer mortar.

In terms of water content in the geopolymer mixture, a number of previous research papers have examined the effect of water content on the workability of fresh fly ash based geopolymer mixtures (Bakharev, 2005b; Hardjito and Rangan, 2005; Davidovits, 2011). Recent research has focused on the final hardened geopolymer products, as water can exist inside in the final products as free or bound water, leading to changes in the geopolymer microstructure (Xie and Kayali, 2014). In order to establish the effect of water content on the compressive strength of geopolymer mortar cured under ambient temperature, where tests were performed on samples with three different water to geopolymer binder ratios of 0.23, 0.25 and 0.28. The effect of water content on the compressive strength of geopolymer are presented in Figure 4.7.



Figure 4.7: Effect of water content on the compressive strength of geopolymer mortar.

The results indicated that increasing the water content did not significantly affect the early strength of geopolymer mortar, but caused a reduction of almost 10% in the 28-days' strength. In the case of Portland cement concrete, water chemically reacts with the cement to bind concrete components together. Water in geopolymer systems only acts as a fluid medium between the dissolved silicates and aluminates ions, providing the required workability (Chindaprasirt *et al.*, 2007). In fact the chemical reaction that occurs in fly ash-based geopolymers produces water that is eventually expelled from the binder, and increasing water content leads to a reduction of the compressive strength of fly ash geopolymer concretes (Vora and Dave, 2013). Xie and Kayali, (2014) noted that lower water content resulted in more compacted structures and higher strength development in ambient temperature-cured geopolymers between the ages of 7 days and 14 days. A reduction in the compressive strength of geopolymer concrete samples cured under elevated temperature with increasing water to solids weight ratio was also observed by (Wallah and Rangan, 2006).

4.3.2.3 Effect of superplasticizer content on the compressive strength of geopolymer mortar.

The compressive strength of geopolymer mortar using different polycarboxylatebased superplasticizer contents are presented in Figure 4.8.



Figure 4.8: Effect of superplasticizer content on the compressive strength of geopolymer mortar.

The addition of super plasticizer showed a significant effect on the compressive strength of geopolymer mortar cured under ambient temperature, particularly at later curing ages. A reduction in compressive strength of around 20% was observed at early age for geopolymer mixtures containing 1% and 1.5% of superplasticizer. This reduction increased with curing age: for example, the compressive strength of geopolymer mortar containing 1.5% superplasticizer reduced by 25% and 32% at 7 days and 28 days, respectively. Increasing the superplasticizer content caused a significant reduction in compressive strength. The compressive strength reduced by 18% and 32% for the geopolymer containing 1% and 1.5% superplasticizer, respectively. However, adding only a small amount of superplasticizer (0.5%) did not show a significant effect on the early and later age compressive strength. Compressive strength reduction due to addition of higher contents of superplasticizer could be due to the fact that the chemically unstable of superplasticizer in multi-compound activator. Nematollahi and Sanjayan, (2014a) examined the impact of a range of different types of superplasticizer on fly ash-based geopolymer, and found that the addition of polycarboxylate-based superplasticizer resulted in a 29% reduction in compressive strength, which agrees with the data presented here.

4.3.2.4 Effect of alkaline activator content on the compressive strength of geopolymer mortar.

Increasing alkaline activator content was found to have a beneficial effect on the compressive strength of the geopolymer mortar. Four different mixtures designed with variant alkali activation to geopolymer binder weight ratios of 8%, 10%, 12% and 14% were examined. Figure 4.9 shows the relationship between potassium silicate (K₂SiO₃) content and compressive strength of the examined fly ash and slag geopolymer mixtures. The effect of curing time was also examined at 3, 7, 14 and 28 days.



Figure 4.9: Effect of alkaline activator on the compressive strength of geopolymer mortar.

Based on the results in Figure 4.9, the compressive strength of the examined mixes increased as the alkaline activator to binder ratio increased from 8% up to 12%. When higher ratios were used (14%), the compressive strength of the examined mixture was reduced. All samples showed an increase in compressive strength with curing age. The compressive strength improvement can be attributed to the formation of calcium silicate hydrate (C-S-H) gel formed with composition and structural differences from those of conventional mortars (Chi, 2012). On the other hand, an excess of alkali solution can increase the water to solid ratio of the mixture leading to higher liquid content which hinders polymerization, and increases the relative content of poorly polymerized reaction products (Nath and Sarker, 2014).

4.3.2.5 Effect of curing conditions on the compressive strength of geopolymer mortar.

Several studies have been carried out to examine the effect of curing temperature on the properties of geopolymer. It was reported that elevated temperature (40 °C to 85 °C) considerably improved the mechanical strength (van Jaarsveld *et al.*, 2002; Bakharev, 2005b; Somna *et al.*, 2011). However, Heah *et al.*, (2011) suggest that curing at elevated temperature for longer curing times caused failure of geopolymer samples at a later age due to the thermolysis of –Si-O-Al-O- bonds. To examine the effect of curing temperature on the hardening properties of user friendly geopolymer mortar the compressive strength results of geopolymer samples cured under elevated temperature have been compared with the respective results of specimens made from the same mix and cured under room temperature (Figure 4.10). The elevated temperature after casting, followed by heat curing at 60 °C for 24 hours, and then storage at room temperature until testing. The resulting compressive strength was measured at 3, 7, 14 and 28 days.



Figure 4.10: Effect of curing temperature on the compressive strength of geopolymer mortar.

At early age, heat curing treatment has a great effect on the strength of fly ash and slag based geopolymer mortar. The 3-day compressive strength of the geopolymer sample cured under elevated temperature is almost double that of the ambient temperature cured sample. This finding is in agreement with the results of a previous study (Part *et al.*, 2015) where curing under elevated temperature was found to be beneficial in

accelerating the dissolution and geopolymerization of the aluminosilicate gel, which results in a high early strength gain. Görhan and Kürklü, (2014) highlighted that curing under elevated temperature leads to an increment in the compressive strength without significant effect on the physical characteristics. It can also be seen that the 28-days compressive strength of the specimens cured under room temperature is very close to the respective results of the heat cured specimens. Based on the observed results of the current study, it could be suggested that elevated temperature curing could be avoided if sufficient curing times are used.

4.3.2.6 Outcomes of Phase I.

When Davidovits, (2011) initially proposed his user friendly system, most of the published studies in this subject area dealt with the use of fly ash or fly ash and slag as source materials for making geopolymer paste, and the resulting chemical and microstructural properties. In addition, the exact details regarding mixture compositions, the manufacturing process and curing conditions were undisclosed. Based on previous studies, a trial and error method was adopted to test different geopolymer materials, and also to generate a successful method with regard to the manufacture of fly ash and slag based geopolymer mortar. Following this, extensive experimental work was performed to examine the effect of the different parameters that influence the fresh and hardening properties of geopolymer mortar. The outcomes of the present study on mix design using a user friendly geopolymer system cured under ambient temperature can be summarised as:

- The sequence of slag addition to the geopolymer mixture considerably effected the fresh and hardening properties of geopolymer mortar. Mixing the solid components first and then adding the liquid solution to the solid blend improved the properties of the final product. However, using prolonged mixing times of up to 23 minutes slightly reduced the compressive strength of geopolymer cured under ambient temperature.
- The inclusion of polycarboxylate-based superplasticizer at up to 1.5% of geopolymer binder by weight improved the setting time and increased the workability. However, this reduces the observed compressive strength.

- The workability and setting time increased as the initial water content was increased from 23% w/b to 28% w/b, while at the same time the compressive strength was reduced.
- Alkaline activator (potassium silicate) content was found to be a crucial parameter for the compressive strength. High potassium silicate content (up to 12% of binder weight) was found to improve the geopolymerization process leading to a more compact structure and strength development.
- Hardened geopolymer materials can be produced without elevated heat curing. The measured compressive strength values of about 40 MPa for fly ash and slag based geopolymer cured under room (ambient) temperature were close to those for materials cured at elevated temperature after 28 days curing. However, elevated temperature significantly improved the early age strength compared with the respective mixture cured under ambient temperature.
- The compressive strength of geopolymer mortar cured under ambient temperature significantly improved with curing age.

To conclude, based on the outcomes of this part of the research study, the mixing procedure and mixture proportion with the optimum fresh properties and compressive strength of fly ash and slag based geopolymer mortar cured under room temperature are listed in Table 4.3.

Item	Weight (Kg/m ³)		
Total Binder	775		
K_2SiO_3	93		
Additional water	194		
Super plasticizer	8		
Sand	1052		
Curing condition	Room temperature		
Mixing procedure	Α		

Table 4.3: Mixing proportion of the optimised geopolymer mortar.

4.4 Phase II. Effect of Slag content on the fresh, hardening and microstructural performance of fly ash and Slag based geopolymer.

The second phase of the current study examined the effect of geopolymer binder composition on the fresh and hardening behaviour of geopolymer mortar. A total of five mixtures were prepared with varying slag to binder weight ratio mixtures (10%, 20%, 30%, 40% and 50%). Based on the results of the first phase, the water content, alkaline activator content and superplasticizer to binder ratio were kept constant at 0.25, 0.12 and 0.01, respectively (Table 4.3). The geopolymer mortar mixtures were proportioned based on previously published work on fly ash based geopolymer mortars e.g. (Wallah and Rangan, 2006). The total binder content was kept constant for all mixtures at 775 kg/m³. The molar ratio of potassium silicate solution (K₂SiO₃used as a chemical activator) MR SiO₂: K₂O remained at the same level for all mixtures at 1.25. The examined mixture compositions for the current study are shown in Table 4.4. Fresh geopolymer mortar properties were examined through workability and setting time tests, while the mechanical performance of the hardened mortar was evaluated by compressive, direct tensile and flexural strength tests. Moreover, the microstructure of the examined mixes was assessed by Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), Fourier Transform Infrared Spectroscopy (FTIR), thermal analysis Differential Scanning Calorimetry (DSC), and porosity measurements.

Mix No.	Mix ID	Fly Ash	Slag	K ₂ SiO ₃ / binder	Water / binder	Polycarboxylate /binder	Mixing Procedure
1	S10	0.9	0.1	0.12	0.25	0.01	Α
2	S20	0.8	0.2	0.12	0.25	0.01	Α
3	S30	0.7	0.3	0.12	0.25	0.01	Α
4	S40	0.6	0.4	0.12	0.25	0.01	Α
5	S50	0.5	0.5	0.12	0.25	0.01	Α

Table 4.4: Mixing proportion of the geopolymer mortar.

4.4.1 Fresh properties of geopolymer mortar with variant slag content.

In this section, two different tests were used to examine the fresh state of geopolymer mortar: setting time and workability. The effect of 10%, 20%, 30%, 40% and 50% slag to binder weight ratios on the setting time of geopolymer mortar is presented in Figure 4.11.



Figure 4.11: Effect of slag content on the setting time of geopolymer mortar.

The results of Figure 4.11 indicate that the setting time was considerably affected by the slag content in the mix. A 100% fly ash mixture was left overnight without any sign of setting, and based on a previous study Jang et al., (2014) the setting time for fly ash based geopolymer without slag has been found to be around 1500 minutes. Setting time was significantly reduced by incorporating slag in the mixture, e.g. the initial and final setting times of mixtures with 10% slag (i.e. the 10S mixture) were reduced to 80 minutes and 150 minutes respectively. As the slag content in the mixture was increased, the total setting time was considerably reduced. The initial and final setting times for the 20S mixture were 58 minutes and 120 minutes while for the 30S mixture these reduced to 42 minutes and 80 minutes. The setting time further slightly decreased when increasing the slag over total binder ratio from 30% to 40%. This is in agreement with previous investigations on the effect of slag content on setting times of fly ash geopolymer mortar with sodium silicate activator. Lee and Lee, (2013) found that the hydration reaction of fly ash based geopolymer materials was accelerated as the slag, and subsequently CaO content, was increased in the mix. Also, Nath and Sarker, (2014) reported that the use of slag resulted in acceleration of the setting time of geopolymer mixes. Flow table tests were carried out to measure the workability of fresh geopolymer mortar.



Figure 4.12: Effect of differing slag contents on the Flowability of the fly ash and slag based geopolymer mortar.

Figure 4.12 shows the Flowability with different replacement ratios of the slag for the fly ash, at 10%, 20%, 30%, 40%, and 50% replacement. The 10S mixture was very fluid and exceeded the flow table limit of 255 mm, while the flow table measurement of the 20S mixture was around 250 mm (Figure 4.12). The workability of the geopolymer mixture was reduced as slag content increased. The reduction in flow was 16%, 30%, and 44% for the 30S, 40S and 50S slag to binder mixtures, compared to the experimental results for the 20S mixture (*e.g.* Figures 4.12 and 4.13). This is likely a consequence of differences in the physical properties and chemical reactions of the mixtures. As the slag content is increased, the number of angular particles is increased and the spherical particles of fly ash are reduced. The spherical shape can help to improve the Flowability of the mortar mixture. Also, admixing high slag content in the fly ash geopolymer increases CaO content, which is the major chemical component of slag (at around 40% by weight), accelerating the geopolymerization process, giving fast setting and reducing the workability.



Figure 4.13: Experimental flow table results for 10S mixture (a), 20S mixture (b), 30S mixture (c), 40S mixture (d), and 50S mixture (e).

4.4.2 Hardening properties of geopolymer mortar with variant slag content.

4.4.2.1 Compressive strength.

The effect of slag content on the compressive strength of geopolymer mortar is presented in Figure 4.14.



Figure 4.14: Effect of slag content on the compressive strength of geopolymer mortar samples cured at ambient temperature.

Figure 4.14 shows the compressive strength of geopolymer specimens with variant slag to binder weight ratios of 10%, 20%, 30% and 40%. Previous studies have indicated that fly ash based geopolymer cured under ambient temperature has very low reactivity (Palomo et al., 1999; van Jaarsveld et al., 2002; Lee and Lee, 2013). As the slag content in the mix was increased, the compressive strength was improved. The three days mean compressive strengths were 2.9 MPa, 10.4 MPa, 13.7 MPa, 18.6 MPa and 25.9 MPa for 10S, 20S, 30S, 40S and 50S mixtures, respectively. At 28 days, mixtures having 20%, 30%, 40% and 50% weight ratio of slag to the total binder achieved 65%, 133%, 140% and 162% higher strength compared to the strength of the 10S mixture, respectively. The results also showed that the effect of slag content is more pronounced at early ages, as the variation between the compressive strength is larger at early curing ages (3 days) rather than at later ages (28 days), especially with higher slag content. This is due to infilling of the porous microstructure of the geopolymer mortar by formation of more hydration products, following the inclusion of the highly reactive slag particles. Shen et al., (2011) examined varying slag/fly ash ratios by weight (0, 30, 40, 50, 60, 70 and 100%), with sodium silicate and sodium hydroxide as the alkaline activator. The main findings of their study indicated that as the slag content was increased, the compressive strength of the mix was improved, in agreement with the work presented here. Buchwald et al., (2009) also indicated that inclusion of slag into a slag and metakaolin based geopolymer accelerated the condensation reaction of alkali activated blends compared to both single phases.

4.4.2.2 Direct tensile test.

Geopolymer tensile strength determination used the briquette tension test. This test method, described in AASHTO T132, (2000), usually includes the direct tension testing of a small briquette cast from mortar. The dog bone-shaped briquette is 25 mm (1 inch) thick, 76 mm (3 inches) long and with a mid-length cross sectional area of 645-mm² (1-inch²). Special self-aligning grips allowed for passive gripping of the specimen in the Instron test machine and ensured uniform loading (Figure 4.15). The briquette tension test used was operated at a constant displacement rate rather than constant load rate, set as 0.4 mm/min.



Figure 4.15: Direct tensile of plain geopolymer set up (a) and dogbone specimen after test (b).

The direct tensile strength of geopolymer mortar mixtures with varying slag content (10–40% wt. of the total binder) cured under ambient temperature was determined at 7 and 28 curing days as shown in Figure 4.16 and Figure 4.17.



Figure 4.16: Stress-strain relationship of plain geopolymer cured under ambient temperature.



Figure 4.17: Effect of slag content on tensile strength of geopolymer mortar samples cured at ambient temperature.

The stress strain behaviour of plain geopolymer considerably improved with increase slag content in the geopolymer mixture. Based on these results, increasing the slag content had a positive effect on the ultimate tensile strength at both curing ages tested. The results indicated that the specimens with 40% slag had the highest direct tensile strength which was around 3 MPa at 28 days. There are no published studies in the literature performing similar tests of direct tensile strength on geopolymer samples, and there are only a few other studies where splitting tensile strength results of geopolymer concrete are presented. Rashad, (2014b) studied the mechanical strength of geopolymer concretes with varying ratios of fly ash to total binder (fly ash and slag) (100%, 95% and 85%) using a mixture of NaOH and sodium silicate as an activator. The results indicated that the split tensile strength was increased as the slag content increased, which is in agreement with the findings of the current study.

4.4.2.3 Flexural strength.

Flexural strength tests were performed according to (ASTM C293-02, 2002) using a simple beam with centre loading with span length (between the supports of the specimens) of 225 mm and an Instron universal test machine (See Figure 4.18b). Load deflection relationships measured by Linear Variable Displacement Transducers (LVDTs) placed on the specimens are shown in Figure 4.18a.



Figure 4.18: Flexural strength testing of plain geopolymer set up (a) and flexural samples after testing (b).

For each mix, three specimens were tested at each age, and the flexural strength is calculated using the average value. Variations in flexural strength with increasing slag content (at curing ages of 7, 14 and 28 days) are shown in Figure 4.19 and Figure 4.20. For each mix, three specimens of each mixture were tested at each age of curing to determine the average flexural strength.



Figure 4.19: Flexural load-deflection relationship of plain geopolymer mortar.



Figure 4.20: Effect of slag content on the ultimate flexural strength of geopolymer mortar samples cured at ambient temperature.

The Figure 4.19 show the load versus deflection for the plain geopolymer mortar. From the curves, the load deflection curve is mostly linear in the pre-peak load region and the peak load improved by increasing slag content. Based on the results, the ultimate flexural strength of geopolymer mortar was considerably increased by increasing the slag content, at all ages (Figure 4.20). At 7 days, the flexural strength of the 10S mix was 0.93 MPa. The flexural strength was improved by 0.6 MPa, 1.3 MPa and 2 MPa as the slag content was increased from 10% to 20%, 30% and 40%, respectively. Flexural strength developed further with longer curing times, with the flexural strength increasing at 14 and 28 days by 100%–245%, 94%– 206%, 48%– 121% and 26%–99% for 10S, 20S, 30S and 40S mixtures respectively, compared to the flexural strength values at 7 days.

4.4.2.4 Porosity measurement.

Porosity measurements were carried out on cylindrical (20mm diameter x50mm length) specimens. The effect of slag content on the apparent porosity of geopolymer mortar cured under ambient temperature for 28 days are presented in Figure 4.21. Two test specimens for porosity measurement were prepared from each mixture.



Figure 4.21: Porosity results for different mixing ratios of geopolymer mortar.

Increasing slag content leads to a reduction in apparent porosity in the geopolymer mortar cured at room temperature (Figure 4.21). Total porosity after 28 days curing of the 10S mixture was around 30%. As the slag content was increased, the porosity was reduced to 23%, 21% and 18.3% for 20S, 30S and 40S mixtures, respectively. The reduction in porosity identified in specimens with higher contents of slag can be attributed to the nature of the gel type forming in the binder. The calcium ions present in slag particles may enter into the fly ash-rich system (with Si-O-Al-O structure) to form a calcium aluminium silicate hydrate gel (C-A-S-H). In addition, unreacted finer slag particles act as a reinforcing agent. This infills the geopolymer matrix, reduces the porosity and improves the mechanical strength.

4.4.2.5 Scanning Electronic Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

Sub-samples taken from 50mm cubes were used to examine sample microstructure after 7 and 28 days. EDS microanalysis on specific sample areas was used to provide semi-quantitative Si, Al, Ca, Na, O, Mg and Fe data from an average of three different areas in the sample, and data for bulk fly ash and slag are given in Table 5 and Figure 4.22.



(a) Fly ash



(b) Slag

Figure 4.22: EDS Analysis of (a) fly ash (top) and (b) slag (bottom).

Element	Slag	Fly ash	
Si	12.3	22.5	
Al	5.1	12.9	
0	38	57.93	
Na	0.3	0.5	
Mg	3.4	0.9	
Ca	37.7	1.0	
Fe	0.4	4.7	

Table 4.5: Atomic weight components (wt. %) slag and fly ash.

As shown in Figure 4.22, the fly ashes consist of spherical particles of various sizes, while the slag is angular and irregularly shaped. Based on the EDS analysis and elemental maps obtained the main contributors to the geopolymer matrix are the glassy fly ash particles (supplying Si and Al), the slag (supplying Ca) and the alkali-activation solution (potassium silicate) (Figure 4.21). To compare the microstructure of different slag to binder ratios of geopolymer, two mixes of 10% and 40% slag to total binder were imaged (Figures. 4.23 and figure 4.24). Figure 4.23 shows the area on a surface of 10% slag at curing ages of 7 days and 28 days at x3000, x10,000 and x20,000 magnification. The micrographs of the 10S mixture show a high proportion of unreacted fly ash spheres, and agglomerated slag particles remaining in the early age matrix. After 28 days curing, the geopolymer microstructure shows amorphous microspheres, and partially reacted fly ash spheres appear in the matrix (Figure 4.23). The loose, relatively uncemented, particle connections observed for geopolymer with low slag content contribute to its low strength. At higher magnification, geopolymer samples show different microstructures with an increasing slag content, which forms a denser matrix than using 10% slag. Glassy crusts covering fly ash particles can be observed, as a result of reactions on the surface of the particles (Figure 4.24 (a–e)). In the geopolymerization method, the development of the geopolymer matrix depends on reactions at the external rim of the particles, producing reaction products which surround and cement the particles, rather than dissolution of the particles (Izquierdo et al., 2009). This clearly corresponds with the strength development results. The presence of calcium in the slag is confirmed by the EDS analysis (at around 38 wt.%), showing that increasing the amount of slag significantly increased the calcium content in the mixture, leading to the formation of a calcium alumino-silicate hydrate (C-A-S-H) gel (Jang et al., 2014). Therefore, inclusion of slag introduced additional calcium
bearing compounds and contributed to additional binding products, which improved the compressive strength, reduced porosity and modified the setting behaviour of geopolymeric gels at early ages. This enabled the formation of a more compact gel structure and consequently improved mechanical properties.





(b) 7 days curing, x10000 magnification





(c) 28 days curing, x3000 magnification

(d) 28 days curing, x10,000 magnification



(e) 28 days curing, x20000 magnification

Figure 4.23: SEM analysis of a 10% slag sample



(e) 28 days curing, x20,000 magnificationFigure 4.24: SEM analysis of a 40% slag sample

4.4.2.6 Fourier Transform Infrared Spectroscopy (FTIR).

Fourier Transform Infrared Spectroscopy (FTIR) is a chemical analysis method which searches for the reaction zones of Si-O and Al-O in order to identify the degree of geopolymerization and formation of reaction products in the various geopolymer mixtures (Ryu *et al.*, 2013). The current study focuses on specific spectral zones: the bands at 1200-900 cm⁻¹ corresponding to the asymmetric stretching vibration of (Si-

O-Si) and (Si-O-Al); the bands assigned around 3350 cm⁻¹ corresponding to the stretching vibrations of H–O–H bonds; and the band around 1645 cm⁻¹ which relates to the bending vibration of the –OH groups of the hydrated reaction products associated with water (Guo *et al.*, 2010; Nath and Kumar, 2013). The functional groups of the raw materials (fly ash and slag), and geopolymer paste containing different slag contents of 10%, 20%, 30% and 40% are presented in Figure 4.25.



Figure 4.25: FTIR analysis of (a) fly ash and slag, and (b) geopolymer paste (10S, 20S, 30S and 40S samples.

The infrared spectroscopic results for the raw materials (fly ash and slag) are presented in Figure 4.25a. The stretching vibration mode of Si-O-T (T represents Si or Al) was found at 903 cm⁻¹ for slag and at 1007 cm⁻¹ for fly ash, which is consistent with (a) the variations in the chemical structure as indicated in the EDS analysis and (b) the glassy structure of these materials. The reduction of the wavenumber of this band for slag corresponds to lower degrees of crosslinking of the amorphous phase of the raw materials, induced by increased calcium content (Rashad, 2014a).

The FTIR spectra of the geopolymer paste samples show clear differences compared to the respective spectra of the fly ash and slag. Figure 4.25b plots the FT-IR results after 28 days for varying slag to binder weight ratios (10%–40%). The Si-O-Si stretching vibration of fly ash occurred at 1007 cm⁻¹. However, the corresponding stretching vibration for geopolymer paste shifted to 959 cm⁻¹, 952 cm⁻¹, 951 cm⁻¹ and 947 cm⁻¹ for 10S, 20S, 30S and 40S mixtures, respectively. Based on these values, increasing the slag content causes structural changes in the examined mixes, which is

attributed to the formation of C-S-H with N-A-S-H type gels and to a subsequent reduction of the amount of Al. These changes lead to accelerated geopolymerization, strengthen the structure of the geopolymer and improve the mechanical strength.

In all geopolymer mixtures, the broad bands present at around 3350–3370 cm⁻¹ are due to the stretching vibrations of H–O–H bonds, while those at 1640–1646 cm⁻¹ are related to the bending vibration of–OH groups in the products of the hydrated reaction associated with water. These bands do not exist in the unreacted fly ash and slag samples and indicate the geopolymerization reaction of alkaline activation products and water into geopolymer pastes.

4.4.2.7 Thermal analysis (DSC).

Thermal analysis was used to evaluate the effect of slag content on the geopolymerisation reaction of the geopolymer paste mixtures using differential scanning calorimetry, within 24 hrs of mixing the dry binder with the alkali activator solution. The DSC curves for the geopolymer mortar with different amounts of slag to binder weight ratios are presented in Figure 4.26.



Figure 4.26: Heat flow vs. time curves for various slag/binder geopolymer mixes, obtained using DSC. All samples contain identical amounts of binder and alkaline liquid.

Isothermal conduction data indicate that the activation process of the geopolymer paste was highly affected by the slag content of the total binder. Thermal power is an indicator of the rate of the chemical reactions occurring between cementitious materials and water and admixtures. The main hydration peak (associated mainly with hydration reactions) is an indicator of setting and early strength development of the mixture (ASTM C 1679, 2008; Lee and Lee, 2015). The heat flow peaks were variable with different slag/binder weight ratio mixtures: the main thermal peaks of the geopolymer pastes were 0.0129 mW/mg, 0.02 mW/mg, 0.0414 mW/mg and 0.034 mW/mg for 20%, 30%, 40% and 50% slag to binder ratios. The initial peak related to the degree of the material particles' reaction and then the heat flow declined to a negative peak followed by small peaks corresponding to the formation of primary C-S-H (Nath and Kumar, 2013). In the case of the 30S mixture, the maximum peaks appeared earlier, possibly because loading the samples in the thermal analyser took a slightly longer time than for the other mixtures. The time to reach the main peaks was reduced as the slag content was increased, although the time between the lower point and the first inflection point in the main maximum peak was increased from 58 to 145 min (by increasing slag from 20% to 50% of the total binder). Yu et al., (2015a) examined the effect of mixing slag and fly ash with Portland cement, and showed that slag can react quickly with Ca(OH)₂ and generate a C-S-H gel, while the generation of this gel is delayed by the reaction of fly ash with portlandite. Therefore, increasing the dosage of slag strongly effects (accelerates) the hydration process of fly ash geopolymer composite, and the time to reach thermal peaks is subsequently reduced. Such a change reduces the setting time of the fresh geopolymer and improves early strength.

The weight loss of the geopolymer samples between the initial sample weight and final weight measurement after removing the samples from the furnace is presented in Table 4.6. The results show that the weight loss increases from 15.5 to 20% by increasing slag content in the binder from 10 to 50%. This loss of weight is an indication of liquid loss and the amount of gel formed during geopolymerization (Guo *et al.*, 2010).

Mix	Slag	Heat flow (mW/mg)			Time (min)		
no.	(%)	Weight loss	Max	Min	Max	Min	Equilibrium
		(%)	peak	peak	peak	peak	
1	10	13.69	0.0419	0.0150	14	72	923
2	20	15.52	0.0129	0.0117	29	87	995
3	30	19.08	0.0200	0.0160	0	288	620
4	40	20.09	0.0415	0.0173	14	159	634
5	50		0.0340	0.0094	14	159	678

Table 4.6: Weight loss and time of heat flow peaks.

4.5 Remarks on the second research phase.

The present study investigated the mechanical and (micro) structural properties of 'user friendly' geopolymer mortars cured under ambient temperatures. These geopolymer mixes were produced using 12% potassium silicate with a 1.25M ratio, which leads to a noncorrosive cement free material. The geopolymer specimens were prepared by dry mixing fly ash and slag at five different ratios in order to optimise the mechanical properties. Fresh geopolymer mortar properties were examined through workability and setting time, while the mechanical performance and physical and chemical characteristics of the hardened mortar were evaluated by compressive, direct tensile, and flexural strength tests, SEM-EDS, porosity and FTIR analysis, and thermal heating. The following conclusions can be drawn from the results outlined here.

4.5.1 Fresh geopolymer mortar characteristics.

• Increasing the slag content in the fly ash-based geopolymer mortar decreases the workability and accelerates the setting times (initial and final) and hardening.

4.5.2 Hardened geopolymer mortar characteristics.

- Compressive strength was considerably affected by blend composition. Improvements in compressive strength have been observed by increasing the slag to total binder ratio in geopolymer mortar mixtures. Increasing slag content from 10% to 50% of the total binder increased the compressive strength from 18.45 to 48 MPa at 28 days.
- Hardened geopolymer materials can be produced without elevated heat curing. The 28-day compressive strength of the specimens cured at room (ambient) temperature is close to the strength of the respective specimens cured under heat treatment.

- The effect of slag content on the development of flexural and direct tensile strengths in ambient temperature cured geopolymer mortar was similar to that observed in the development of compressive strength. The 40S mixture showed optimum results for flexural and tensile strength, at 6 MPa and 3 MPa respectively.
- The addition of slag lowered the total porosity of the fly ash and slag based geopolymer mixture.
- SEM/EDS analysis enabled assessment of microstructural evolution and reaction
 product formation in the geopolymer samples. The images show a less dense
 structure and non-reacted fly ash particles with low contents of slag. However, the
 compactness of the geopolymer matrix increased when slag content was increased.
- FTIR analysis allowed assessment of the degree of geopolymerization and the formation of reaction products. The addition of slag affected the structural reorganisation by increasing C-S-H gel formation and reducing the amount of aluminosilicate gel related to fly ash, which in turn accelerates geopolymerisation.
- Thermal analysis (DSC) showed that the addition of slag increased the heat flow in the samples and reduced the time to achieve the main thermal (reaction) peak due to accelerated formation of cementitious gels, leading to a reduction in the setting time.

The overall conclusion of the current study is that the examined 'user friendly' geopolymer mixes with increased slag content had considerably improved flexural and direct tensile strength, even without any heat curing treatment, which makes the proposed method suitable for in situ applications. Further improvement of the mechanical properties of the examined mixes could be achieved by the addition of silica fume, fibres or nanoparticles (Saafi *et al.*, 2013).

CHAPTER 5:

DEVELOPMENT OF TERNARY PLAIN GEOPOLYMER MORTAR AND STEEL FIBRE REINFORCED GEOPOLYMER CONCRETE

5.1 Introduction.

Most previous studies on fly ash-based geopolymer cured at ambient temperature (i.e. for cast-in-place concrete applications) highlight its relatively poor early strength development due to a slow polymerisation process (Adak et al., 2014). The key factors affecting the potential reactivity of fly ash include the vitreous phase content, reactive silica content, and the particle size distribution (Olivia and Nikraz, 2012; Riahi and Nazari, 2012; Chi and Huang, 2013). Therefore, researchers have attempted to enhance the reactivity of fly ash-based geopolymer by reducing the fly ash particle size, or by adding quantities of calcium containing materials to react with the fly ash particles. Inclusion of ground granulated blast slag (GGBS) as source of calcium together with fly ash has been investigated with favourable results, as detailed in Chapter Four. Reactivity can also be increased by addition of ultra-fine particles of amorphous silica, or Silica Fume (SF), which are available commercially in various forms depending on the material handling techniques (i.e. as densified, undensified and water-based slurries), and have been shown to improve the mechanical properties of both high performance and conventional concretes. For example, the addition of silica fume during the production of high strength concrete (HSC) has been observed to improve interfacial cement paste-aggregates bonding, which is the weakest zone in the matrix (Köksal et al., 2008; Güneyisi et al., 2012). Ivorra et al., (2010) studied the effect of silica fume particle size distribution on the performance of Portland cement mortar, and observed that the strength of conventional concrete was improved by using SF with finer particles. This was attributed to the enhanced filler effect of finer SF, and to higher pozzolanic reactivity due to an increased specific surface area.

Using pozzolanic materials such as silica fume, ground granulated blast-furnace slag (GGBFS), and fly ash (FA) as a replacement for conventional OPC can however cause deterioration in some key mechanical properties. For example, use of these materials can increase brittleness, and cause development of cracks over time due to plastic

shrinkage in the pre-hardened state, as well as drying shrinkage in hardened concrete (Afroughsabet and Ozbakkaloglu, 2015). These cracks reduce the material durability and subsequently its service life. Limiting brittleness and crack propagation, while at the same time improving the early strength and reactivity of geopolymer materials, is therefore of key importance for the development of effective geopolymer materials which can be cured or produced under ambient (i.e. on-site) temperatures. It is well-known that brittleness and cracking effects can be mitigated by the addition of fibre reinforcements into the matrix, which control the propagation or coalescence of cracks (Kim *et al.*, 2008), and reduce the tendency for brittle material failure. Steel fibres are commonly used for reinforcing conventional concretes in this way, and are manufactured from cold-drawn wire, steel sheet and other forms of steel (Soutsos *et al.*, 2012).

The main improvements in the engineering properties of the concrete following inclusion of fibres are strain hardening after the peak load, fracture toughness, and resistance to fatigue and thermal shock (Soutsos *et al.*, 2012). To date, however there is a very limited literature on fibre reinforced geopolymer concrete (Bernal *et al.*, 2010; Shaikh, 2013a), and this focuses on geopolymer materials hardened under heat curing conditions with single/ binary geopolymer binders. There is no published study examining the impact of different geopolymer matrix compositions and silica fume forms on the fresh properties, mechanical and microstructural characteristics of plain and steel fibre reinforced geopolymer concrete (SFRGC) cured under ambient temperatures. It is essential to evaluate the influence of these parameters on the matrix and composite properties with the aim of selecting an appropriate type of matrix for cast-in-place applications.

The previous chapter discussed the fresh, mechanical and microstructure properties of binary blended slag and fly ash based geopolymer without steel fibre reinforcement. The work presented in this chapter addresses thesis objectives 1 and 2. The effect of primary geopolymer material composition and geopolymer matrix strength on the bond performance of plain and steel fibre reinforced geopolymer concrete (SFRGC) cured under ambient temperatures has been examined by using a varying ternary geopolymer mixture (fly ash, slag and silica fume, mixed with potassium silicate as an alkaline solution). An extensive experimental study has been conducted to examine

the influence of slag content and variant silica fume (SF) forms on the fresh, mechanical and microstructural properties of plain geopolymer mortar and SFRGC.

5.2 Experimental procedures.

Thirty-two different mixes (Table 5.1) were prepared to evaluate the effect of ground granulated blast slag (GGBS) content and silica fume particle size distribution on the mechanical behaviour of plain geopolymer and steel fibre reinforced geopolymer composites (SFRGC). 10% of the fly ash content was replaced by dry silica fume (densified silica, undensified silica) (in samples with suffix "DSF" or "USF"), whereas 5% of fly ash content was replaced in samples containing slurry silica fume (samples with suffix "SSF"). Slag was also added at varying slag to binder weight ratios, of 10%, 20%, 30% and 40% (samples with prefix 10S, 20S, 30S and 40S respectively), while steel fibre was added at 2% volume fraction. Reference geopolymer mortar specimens with similar slag to binder weight ratios of 10%, 20%, 30% and 40%, with silica fume but without steel fibre (ST) were prepared as controls, to allow assessment of the impact of ST on material performance.

Mixture ID	Slag	Silica fume	Slag	Silica	Fly Ash	Steel
	· ·	type	C	fume	·	Fibre
	[%]		[kg/m ³]	$[kg/m^3]$	[Kg/m ³]	$[V_f]$
10S/10S-ST			78	0	698	0/2%
10S-10DSF/10S-10DSF-ST	10%	densified	78	78	620	0/ 2%
10S-10USF/ 10S-10USF-ST		undensified	78	78	620	0/ 2%
10S-5SSF/ 10S-10SSF-ST		slurry	78	39	659	0/ 2%
20S/20S-ST			155	0	620	0/ 2%
20S-10DSF/20S-10DSF-ST	20%	densified	155	78	543	0/ 2%
20S-10USF/ 20S-10USF-ST		undensified	155	78	543	0/ 2%
20S-5SSF/ 20S-10SSF-ST		slurry	155	39	581	0/ 2%
30S/30S-ST			233	0	543	0/ 2%
30S-10DSF/30S-10DSF-ST	30%	densified	233	78	465	0/ 2%
30S-10USF/ 30S-10USF-ST		undensified	233	78	465	0/ 2%
30S-5SSF/ 30S-10SSF-ST		slurry	233	39	504	0/ 2%
40S/40S-ST			310	0	465	0/ 2%
40S-10DSF/40S-10DSF-ST 409/		densified	310	78	388	0/ 2%
40S-10USF/ 40S-10USF-ST	40%	undensified	310	78	388	0/ 2%
40S-5SSF/ 40S-10SSF-ST		slurry	310	39	426	0/ 2%

Table 5.1: Mixture proportioning of the plain geopolymer mortar and SFRGC used in the present study. See text for discussion of mixture ID notation.

All geopolymer mortars were mixed using a 5 litre Hobart mixer. The mixing procedure proposed by GEOASH, Davidovits et al., (2014) was used in this study. The alkaline solution was prepared by dilution of potassium hydroxide pellets with distilled water in a fume cupboard. The solution was left for 24 hours to cool down to room temperature before mixing with potassium silicate solution (mass of Potassium hydroxide solution to Potassium silicate solution was equal to 2.5), to form a solution modulus equal to 1.25. For geopolymer mortar mixtures with dry powder silica fume (Densified and Undensified), the liquid phase including potassium silicate solution was prepared in advance and mixed with water and superplasticizer 5 minutes prior to mixing with the solid phase. The binder powder materials (fly ash, slag and silica fume) were dry mixed for 5 minutes at low speed (140±5r/m) to ensure adequate mixing. The liquid phase was then added to the solid phase and the mixer run at medium speed for 5 minutes. After that, steel fibres were slowly added to the mixture. Finally, sand was added to the mixer, and the mixer was run for a further 3 minutes to give a total mixing time of 13 minutes (Figure 5.1). However, in the case of mixes with slurry silica, the mixing step was changed, with the sand added before the slurry silica. This revised mixing procedure was necessary to avoid flash setting, as the high reactivity of the slurry silica can lead to gelation of the geopolymer binder without sand (Figure 5.1).



Figure 5.1: Mixing procedure of SFRGC with different silica fume forms.

The fresh geopolymer was immediately cast into moulds. After 24 hours, all specimens were removed from their moulds and covered with plastic film to avoid evaporation of water, then stored under room (i.e. ambient) temperature for curing (21-23°C) until the day of testing. The fresh properties of the geopolymer mortar were examined via setting time and workability analysis. The initial and final setting times of plain geopolymer mortars without steel fibres were measured using a vicat needle according to BS EN 480-2, (2006). Setting time tests were conducted at room

temperature (21-23 °C). Flow tests were undertaken immediately at the end of the mixing based on ASTM C230, (2002)). The mechanical performance of hardened plain geopolymer mortar and SFRGC was also examined to establish the optimum slag percentage and silica fume form in compressive strength and direct tensile strength tests, and energy absorption tests. The microstructure of the examined composites was assessed by particle size analysis, scanning electronic microscopy (SEM) and porosity measurements.

5.3 Results and discussion.

5.3.1 Characterization of Silica Fume.

Various types of silica fume with different physical properties were utilised in this study. The densities and specific gravity for all silica fume forms as received from the manufacturer are shown in Table 5.2. Particle size distribution was determined using a Malvern Mastersizer 2000 laser diffraction particle size analyser (Figure 5.2 and Table 5.3).

Table 5.2: Bulk density of silica fume types (as received from the manufacturer).

Silica type	Bulk density (kg/m ³)		
Undensified silica (USF)	130-430		
Slurry silica (SSF)	1320-1440		
Densified silica (DSF)	480-720		

Table 5.3: Particle size analysis data for densified silica fume (DSF), undensified silica fume (USF) and slurry silica (SSF).

Particle Size (µm)	DSF (µm)	USF (µm)	SSF (µm)
d(0.1)	36.4	4.3	0.1
d(0.5)	203.6	37.1	0.3
d(0.9)	428.8	126.7	1.5



Figure 5.2: Particle size distribution of SSF (a), DSF and USF (b), and Fly Ash and GGBS (c).

Table 5.3 shows the most common statistics used to analyse the particle size: the mean particle size (d(0.5)), and d(0.1) and d(0.9) (the particle sizes where 10% and 90% of the sample population are smaller than this size). The aqueous suspension (slurry) with a dry silica fume content of 50% by mass (SSF) showed the smallest particle size followed by undensified silica fume (USF) and finally densified silica fume (DSF). These results are due to particle agglomeration during the production and packaging procedure of the silica fume. During the formation of silica fume at high temperature (>1000°C), primary particles condense and are bound immediately to clusters of several spheres by sintered junctions through Si–O–Si bonds. Agglomerates of clusters form both when the material cools and is stored in the silo (i.e. undensified silica fume as used in this study) or in the air densification process (to produce densified silica fume).

5.3.2 Fresh characteristics of geopolymer mortar

The fresh properties of geopolymer mortar were examined by setting time and workability. The workability of geopolymer mortar is largely determined by water content, or wetness of the mixture, prior to casting. The particle size distribution and chemical components of the geopolymer mortar also affect the fresh characteristics. The effect of slag content and silica fume particle size distribution on the fresh properties of plain geopolymer mortar are shown in Figure 5.3.



Figure 5.3: The fresh properties of geopolymer mortar; 10% slag/ binder mixtures (a), 20% slag/ binder mixtures (b), 30% slag/ binder mixtures (c) and 40% slag/ binder mixtures (d).

Based on these results, the workability of the mixes is considerably affected by the geopolymer binder composition. The results indicated that increasing slag content in the geopolymer mortar considerably decreases the workability (Figure 5.3). The workability of the geopolymer mortar is reduced from 250 mm to 210mm, and 175 mm as slag is increased from 10% to 30% and 40%, respectively. However, increasing the slag content from 10% to 20% did not show a significant effect on the workability results. This reduction in workability is attributed to the enhanced reactivity of the fly ash-slag mixture, and in particular the rapid reaction between the geopolymer binder and the alkaline solution (potassium silicate). The incorporation of ultra-fine silica fume (USF and SSF) in the mixtures also reduces the workability compared with the respective control geopolymer mixtures (without silica fume). The workability of the 10S mixture reduced from 250 mm to 230 mm, and 220 mm by inclusion of 10USF

and 5SSF, respectively. Similar trends were observed in the mixtures containing higher slag content, where the workability of the 40S mixture was reduced by 9% and 20% by inclusion 10USF and 5SSF, respectively. Since very fine silica fume particles have large effective surface area, they rapidly adsorb water and thus the workability of the geopolymer mortar is reduced. However, the workability of the geopolymer mixtures was not significantly affected by inclusion of large particle sizes of silica fume (DSF).

It is also evident from the results presented in Figure 5.3 that both initial and final setting times are considerably reduced as the slag content is increased from 10% to 40%. The initial and final setting time reduced by 28% and 20% for the 20S mixture, 48% and 36% for the 30S mixture, and 56% and 50% for the 40S mixture compared to the respective setting times of the 10S mixture. This is attributed to the increment in CaO content, which is the main chemical component of slag, and the subsequent acceleration of hydration reactions. The incorporation of silica fume in the mixture also affects both initial and final setting time for all the examined mixtures. The initial and final setting times were reduced when undensified (USF) and slurry silica fume (SSF) were added to the mix (a trend seen most clearly in the lower slag content samples). The initial and final setting time reduced from 80 min and 150 min for the binary 10S mixture to 55 min and 135 min for the 10S-10USF mixture and 45 min and 115 min for the 10S-5SSF mixture, respectively. However, inclusion of DSF in the mixture prolonged the initial and final setting time to 87 min and 152 min for the 10S mixture, respectively. This contrasting behaviour is related to the finer particle size and higher surface area of the USF and SSF compared to densified silica fume (DSF), which accelerates the geopolymerization process and hardening of the geopolymer mortar. At higher slag content mixtures, similar trends are observed as the initial and final setting time was reduced by inclusion of fine silica fume however, the reductions in the setting time were less pronounced than in low slag content mixtures.

5.3.3 Mechanical Properties

5.3.3.1 Porosity Measurements

Sample porosity was measured through the vacuum saturation technique, as reported in previous studies (Gonen and Yazicioglu, 2007; Keleştemur and Demirel, 2010) and described in detail in Chapter Three. The effect of slag content and silica fume particle size on the apparent porosity of geopolymer mortar is shown in Figure 5.4. Porosity tests were carried out on at least two cylindrical samples (20mm diameter and 50mm length) for each mixture.



Figure 5.4: Porosity (%) of ternary geopolymer mortar with variant forms of silica fume.

A clear reduction of porosity values is observed as the slag content is increased, as the reaction of slag with fly ash causes formation of more hydration products, which leads to a denser matrix. The inclusion of silica fume also plays an important role in controlling the porosity of geopolymer mortar at low % slag content. The total porosity of geopolymer mortar containing undensified and slurry silica fume particles is considerably lower than the control mixture (without silica fume) for the 10S mixture. However, this effect is not observed in mixes with higher slag content. The increment of the slag content in the geopolymer binder from 10% to 40% leads to an improvement of geopolymer matrix reactivity which is sufficient to reduce the effect of inclusion of undensified and slurry silica fumes on the total porosity. As noted above, inclusion of slag as highly pozzolanic material increases the geopolymerization products and produces a denser matrix. In contrast, the results clearly indicate that the total porosity of the geopolymer mortar is increased with inclusion of larger particle sizes of silica fume (DSF), for all mixes. The total porosity of the 10S mixture decreases from 30% to 23.5% and 25.31% by the addition of USF and SSF, while it increases to 31.2% using DSF. Therefore, while the use of undensified and slurry silica tends to reduce porosity, as they compact the matrix by filling pore spaces and improving reactivity and bonding at the geopolymer paste-silica sand interfacial zone (Ivorra et al., 2010; Güneyisi et al., 2012), densified silica fume (which is much

coarser) has a much lower reactivity, negative pore filling effect, and subsequently causes increased porosity.

5.3.3.2 Compressive strength test.

Previous research has indicated that addition of slag and silica fume to ordinary Portland cement leads to improved strength characteristics (Oertel *et al.*, 2014). Ultrahigh performance concretes are manufactured by mixing high amounts of cement, slag and silica fume with low contents of water (Yu *et al.*, 2014). This is due to the lower lime content of slag blended cements causing an initially slow hydration and the production of more C-S-H as a result of the reaction, resulting high strength concrete. There are some studies examining binary blended mixes for producing geopolymer however, the role of ternary blended fly ash, slag and silica fume on the mechanical properties of user friendly geopolymer mortar cured under room temperature has not been examined. Compressive tests were therefore carried out to evaluate the strength characteristics of (a) hardened plain geopolymer mortar, and (b) steel fibre reinforced geopolymer composites, based on a ternary blended mix. Compressive strength tests were conducted according to ASTM C109 . Three cubic specimens with 50 mm sides were used to determine compressive strength of geopolymer samples at 3, 7, and 28 days.

5.3.3.2.1 Compressive strength development of plain ternary geopolymer mortar.

The compressive strengths of each plain geopolymer mixture with different slag replacement content and silica fume forms at 3, 7 and 28 days are shown in Figure 5.5.





Figure 5.5: The compressive strength of plain geopolymer mixes containing binary and ternary mixes at 3 days, 7 days and 28 days, cured at ambient temperatures. Error bars are shown for n=3.

The results in Figure 5.5 showed that the compressive strength of plain geopolymer mortar increased with increasing slag content for all mixtures, at both early and late stage curing. This improvement in the compressive strength is due to an increase in the calcium content in the mixture, which leads to infilling of the porous microstructure of the geopolymer composite by the formation of more hydration products from both slag and fly ash (as discussed in Chapter Four).

The binary blended geopolymer mixture containing varying slag to binder weight ratios was used to provide a benchmark, or control, against which the ternary geopolymer mixtures (fly ash, slag, and silica fume) could be compared. At early age (3 days), the results in Figure 5.5 showed that utilization of densified silica fume negatively affected compressive strength at all slag contents. Inclusion of Undensified (USF) and slurry (SSF) silica fume however enhanced the compressive strength, by 50%-74%, 17.4%-18.6%, 66.4%-50.2%, and 56.7%-16.3% for 10S, 20S, 30S and 40S mixtures, respectively. This improvement in the compressive strength relates to the fine particle sizes of undensified (USF) and slurry (SSF) silica fume, with high specific surface area leading to acceleration of the geopolymerization of aluminosilicate gel, which leads to high early strength gain and compacts the matrix structure by filling pore spaces. In contrast, the agglomerated particles of densified (DSF) silica fume have larger size and lower pozzolanic activity, leading to a more porous microstructure and loss of strength. The dispersion of silica into smaller primary particle sizes or the smallest agglomerates possible is a crucial parameter for further improvement of the compressive strength. At later ages (7 days and 28 days), similar trends were observed and ternary geopolymer mixtures containing USF showed the highest compressive strength values followed by SSF mixtures and control binary blended mixtures.



Figure 5.6: (a) Geopolymer cubic with undensified, slurry and densified silica fume (b) fractured geopolymer specimens after compressive strength test.

5.3.3.2.2 Compressive strength development of SFRGC mixtures.

In order to examine the effect of different geopolymer matrix compositions on the compressive strength of steel fibre reinforced geopolymer mixture. Different slag content and silica fume forms on the compressive strength of SFRGC material have been examined. For each mixture, twelve cube specimens of SFRGC were tested at 3, 7, 14 and 28 days.



Figure 5.7: The compressive strength of SFRGC mixes containing binary and ternary mixes at 3 days, 7 days and 28 days curing at ambient temperatures. Error bars are shown for n=3.

Figure 5.7 shown the outcomes of compression tests on SFRGC specimens with varying silica fume forms, at different curing ages. In all SFRGC composites, the compressive strength increased with increased slag to total binder weight ratio. Moreover, it was observed that compressive strength values of SFRGC mixtures showed diverse performance by inclusion of variant silica fume forms.

At 10% slag to binder weight ratio, the binary (fly ash and slag) geopolymer mixture was higher in strength than the ternary (fly ash, slag and silica fume) geopolymer mixtures in the case of USF and SSF. However, using DSF form in the SFRGC composite slightly reduced the compressive strength compared to the binary mixture containing same slag content. When 20%- 40% slag to binder weight ratios were added to the geopolymer mixture this had considerable effect on the strength development. The compressive strength of SFRGC developed from 18 MPa for the 10S-2ST mixture to 28 MPa, 47 MPa, and 50 MPa for the 20S-2ST, 30S-2ST, and 40S-2ST mixtures, respectively. The effect of silica fume on the ternary SFRGC composites containing 20% and 30% slag to binder weight ratio showed a similar trend to the lower slag content mixtures. The compressive strength of the SFRGC mixture increased by 10%, 36%, and 13% for 20S-10DSF-ST, 20S-10USF-ST, and 20S-5SSF-ST mixtures compared to the respective strength value of the 20S-ST mixture. This difference was less pronounced at 40% slag to binder weight ratio mixtures apart from for the USF form. The highest compressive strength was found in the 40S-10USF-ST mixture, at 64 MPa.

5.3.3.2.3 Effect of curing time on the compressive strength development of plain geopolymer and SFRGC.

The effect of curing time on the compressive strength development of plain geopolymer mortar and SFRGC mixtures are shown in Figure 5.8 and Figure 5.9.



Figure 5.8: Strength versus curing time relationship of plain geopolymer.



Figure 5.9: Strength versus curing time relationship of SFRGC.

Figure 5.8 shows a comparison of compressive strength development of geopolymer mortar cured under ambient temperature over curing times of 3 days, 7 days and 28 days. The results indicated that as the age of geopolymer specimens increase from 3 days to 28 days, compressive strength significantly improved for all the examined mixtures. At 10% slag to binder wt. ratio mixture, the 10S mixture specimens gained 16% and 46% of its 28 days compressive strength at 3 days and 7 days, respectively. The results also showed an enhanced impact of the inclusion of silica fume at early

ages for the plain geopolymer mortar. The 3 days compressive strength of 10S ternary geopolymer mixtures increased by 50% and 74% for USF and SSF, respectively and reduced by 56% for DSF compared to the binary geopolymer mixture. While, at 28 days the compressive strength of 10S ternary geopolymer mortar mixtures increased by 17% and 8% for USF and SSF, respectively and reduced by 14% for DSF compared to the binary geopolymer mixture. The early and later age strength of geopolymer mortar containing low slag content were very low compared with the respective strength values of geopolymer mortar containing high slag content. For instant, the compressive strength of the 40S-10USF mixture increased from 29 MPa to 38 MPa and 45 MPa at 3 days, 7 days and 28 days, respectively. A similar trend was observed in the steel fibre reinforced concrete (Figure 5.9), where the early age compressive strength was significantly lower than the later age compressive strength. The compressive strength of 20S-10USF-ST and 40S-10USF-ST mixtures increased from 12.4 MPa and 30 MPa at 3 days to 22 MPa and 41 MPa at 7 days and 32 MPa and 64 MPa at 28 days, respectively. When geopolymer specimens are cured under room temperature, the geopolymerization process considerably improves with sufficient curing time, leading to improvement in strength of the geopolymer matrix, and enhancement of the bonding between the geopolymer matrix and the reinforcement fibre. Thus, a high strength geopolymer mortar cured under ambient temperature was achieved by using a ternary binder (fly ash, slag and silica fume) mixed with 2% steel fibre.

5.3.3.2.4 Comparison of the compressive strength of plain geopolymer mortar with SFRGC mixes.

The influence of steel fibre addition on the compressive strength of geopolymer mixes (at 28 days curing) is shown in Figure 5.10



Figure 5.10: Percentage increase in 28 days compressive strength of SFRGC over that of plain geopolymer mortar.

In all SFRGC composites, addition of steel fibres to the geopolymer samples had a clear impact on compressive strength development in the geopolymer. Steel fibre addition reduced the compressive strength of the lower strength geopolymer matrix with 10% and 20% of slag to binder weight ratios. At higher slag content, however the compressive strength of SFRGC was significantly improved compared to the respective mixes without steel fibres. The low strength values observed for SFRGC mixes with lower slag content are apparent from early curing ages onwards, and reflect reduced pozzolanic activity (and reactivity) and lower reaction product formation, which weakens the interfacial bonds between the steel fibre and the geopolymer matrix.

When considering the effect of combining steel fibre with different silica fume forms, the compressive strength of steel fibre reinforced geopolymer composites with incorporated silica fume was equivalent to or higher than the plain geopolymer, with the exception of samples with very low slag content (10% slag). Inclusion of silica fume in the SFRGC mixture with 20% slag content increased the compressive strength by 18%, 9% and 5% for DSF, USF and SSF, respectively. However, the compressive strength is reduced by 8% for the 20% slag mixture without silica fume. At high slag content, the compressive strength of steel fibre reinforced geopolymer composite with USF showed the highest recorded strength value at around 63 MPa (40% slag

mixture), and SFRGC samples containing USF showed the largest and most consistent increase in compressive strength over the control SFRGC (i.e. without added silica fume). Moreover, the failure mode of all SFRGC specimens changed from brittle to ductile due to the bridging effect of steel fibres, and the samples kept their original shapes up to the end of the test (Figure 5.11).



Figure 5.11: cubic specimens after compressive strength testing: plain geopolymer (a), and SFRGC (b).

5.3.3.3 Direct tensile test of SFRGC

It is well known that the most suitable, and physically sound, classification of conventional concrete tensile behaviour is based on the uni-axial response (Li *et al.*, 1993). Direct tensile strength tests were carried out to evaluate the effect of silica fume forms and slag content on the behaviour of SFRGC under tension. Direct tensile tests were conducted under displacement control with a constant rate of 0.4 mm/min using an Instron testing machine (Hassan *et al.*, 2012), using the testing configuration shown in Figure 5.12. The average extension was measured using Linear Variable Displacement Transducers (LVDT) attached to a steel frame as shown in Figure 5.12b.



Figure 5.12: SFRGC specimen (a) and direct tensile strength test set up (b).

The tensile strength behaviour of conventional steel fibre reinforced concrete can be classified as either pseudo strain hardening composite or strain softening composite (a quasi-brittle material) (Figure 5.13). The tensile classification of each mixture can be clarified from the stress strain relationship. However, the performance of the material under tensile stress depends on the strength of the cementitious matrix, and the fibre reinforcement type and aspect ratio. In this section, the direct tensile strength results of steel fibre reinforced geopolymer, focusing on the effect of the geopolymer matrix, are presented. In addition, the elastic modulus, ultimate post-cracking tensile stress, and the energy absorbed by the specimens (the area under the tensile stress-strain curve) were analysed and investigated.



Figure 5.13: Typical tensile behaviour of steel fibre reinforced concrete.

5.3.3.3.1 Tensile stress-strain relationship of SFRGC over the curing time.

In order to evaluate the tensile strength development of steel fibre reinforced geopolymer cured under ambient temperature, twelve dog bone specimens for each mixture were examined under tension at 3 days, 7 days, 14 days and 28 days (Listed in Table 5.4). In addition, the effect of curing time on the stress strain relationship of SFRGC was examined using the 40S-10USF-ST mixture as an example and the average of stress versus strain (extension normalized to the gauge length) results of all the 6 specimens for each mixture are presented in (Figure 5.14).





Figure 5.14: Tensile stress-strain curve of Ternary SFRGC mixtures with USF and different slag contents at 3 days, 7 days, 14 days and 28 days.

Mix No.	Tensile Strength (MPa)					
	3days	7days	14days	28days		
10S-ST	0.22	0.30	0.54	0.60		
10S-10DSF-ST	nm	nm	0.65	0.96		
10S-10USF-ST	0.36	0.41	1.15	2		
10S-10SSF-ST	0.42	1.92	2.14	2.22		
20S-ST	0.33	0.61	0.69	1.36		
20S-10DSF-ST	nm	0.27	nm	1.46		
20S-10USF-ST	0.59	0.94	1.32	2.39		
20S-10SSF-ST	0.25	0.51	0.65	1.60		
30S-ST	0.54	0.58	1.64	2		
30S-10DSF-ST	0.34	1.05	NA	2.20		
30S-10USF-ST	0.589	1.33	1.75	2.60		
30S-10SSF-ST	0.51	0.81	1.12	1.40		
40S-ST	0.96	1.35	nm	2.80		
40S-10DSF-ST	0.424	1.406	1.70	2.80		
40S-10USF-ST	1.10	1.43	2	3.10		
40S-10SSF-ST	1.30	1.60	1.67	2.30		

Table 5.4: Tensile Strength of steel fibre reinforced geopolymer concrete over curing age.

*nm: not measured, due to failure to test within target date

The results indicate that curing time plays a major role in the strength development of steel fibre reinforced geopolymer concrete under ambient temperature. The data in Table 5.4 showed that increasing the curing time gives higher strength compared to the respective values at early age for all geopolymer mixtures (see Figure 5.14 for all slag content mixtures of SFRGC with USF as an example). The tensile strengths for samples cured under ambient temperature at the age of 3 days for 10S, 20S, 30S and 40S mixes were 0.2 MPa, 0.3 MPa, 0.54 MPa and 0.96 MPa, respectively. For a curing duration of 7 days, the strength were 0.3 MPa, 0.6 MPa, 0.58 MPa and 1.35 MPa and for a curing duration of 28 days, the strengths were 0.6 MPa, 1.36 MPa, 2 MPa and 2.8 MPa, respectively. Similar trends were observed in the strength development of ternary geopolymer mixtures under ambient temperature. The 10S-10USF-ST mixture specimens gained 18%, 20% and 55% of their 28 days tensile strength at 3 days, 7 days and 28 days, respectively. By comparison, the 40S-10USF-ST mixture specimens gained 35%, 46%, 65% of their 28 days tensile strength at 3 days, 7 days, and 14 days, respectively. The mixtures with high percentages of low pozzolanic reactive (fly ash) material need more time in order to produce more geopolymerization products, increase the compressive strength of the geopolymer matrix and improve the bond

between the steel fibres and the geopolymer matrix. Figure 5.13 indicates that curing time also significantly effects the stress-strain relationships of SFRGC mixtures. Increasing the curing time increased the stress-strain relationships for all the examined mixtures and this is clearly observed in the elastic state and absorption energy (See Figure 5.15 and Figure 5.16).



Figure 5.15: Modulus of elasticity of SFRGC over curing time.



Figure 5.16: Fracture energy of SFRGC over curing time.

The elastic modulus defines the linear part of the stress-strain curve up to the nonlinear stage, which represents the first cracking of the material. From the results presented in Figure 5.15, the Young's modulus of elasticity significantly increased with curing time for all of the examined mixtures. The modulus of elasticity of 30S-10USF-ST

increased from 3900 MPa at 3 days to 4500 MPa, 5200 MPa, and 7500 MPa at 7 days, 14 days, and 28 days, respectively. The energy absorption capacity of the SFRGC up to 2% strain capacity after appearance of cracking also confirms the effect of curing time. The energy absorption of the 40S-10USF-ST specimens increased by 58%, 130%, and 220% at 7 days, 14 days, and 28 days respectively compared to their respective energy absorption values at 3 days.

5.3.3.2 Tensile stress-strain relationship of SFRGC with variant silica fume forms and slag content.

In order to provide a basis of comparison and for control, the direct tensile strength of plain geopolymer mortar was investigated using a small dog bone specimens as described in Chapter 4. The tensile stress-strain behaviours for the plain geopolymer mortar and steel fibre reinforced geopolymer composite mixtures with variant slag contents are shown in Figure 5.17 and Figure 5.18, respectively. An average stress strain curve was selected from each series of tests and used for comparison.



Figure 5.17: Stress-strain relationship of plain geopolymer.



Figure 5.18: Effect of slag content on the stress-strain curve of SFRGC.

Based on the observed stress-strain relationships, the geopolymer binder composition and the addition of steel fibre significantly affects the tensile behaviour of the samples. The geopolymer binder composition (slag content) impacts on the precracking behaviour (elastic) part of the graphs and the extent to which this ensures effective transfer of stress to the fibres. The linear part is followed by the non-linear plastic state which is a function of the postcrack behaviour at the steel fibre and geopolymer matrix interface. The geopolymer concrete reaches the peak load through non-linear strainhardening followed by strain softening once micro-cracking increases. The addition of steel fibres in the geopolymer composite considerably improves the post-crack load carrying capacity due to the reinforcing effect of the steel fibres in all examined mixtures which leads to change in the failure mode from fragile (i.e. sudden failure) to ductile failure, due to the bridging effect of the fibres and reduced crack coalescence.

The results show that the peak stress and post crack behaviour of SFRGC mixtures with low slag content were poor due to weak bonding between the low strength geopolymer matrix and the steel fibres. However, as slag content was increased in all the examined mixtures, the the post crack carrying capacity was improved (Figure 5.18). This is due to higher slag content increasing reaction products such as C-S-H,

C-A-S-H and N-A-S-H (Al-Majidi *et al.*, 2016b), which in turn strengthens the interfacial transition zone with the steel fibres within the geopolymer matrix.

In order to evaluate the effect of ternary blended geopolymer mixtures on the tensile performance of steel fibre reinforced geopolymer composite, tensile stress-strain results of the examined mixtures with different silica fume forms (DSF, USF, and SSF) were compared with binary control mixture (fly ash and slag) for all slag contents, and are presented in Figure 5.19.





Figure 5.19: Effect of silica fume forms and slag content on the stress-strain relationship of SFRGC; 10% slag/binder mixtures (a), 20% slag/ binder mixtures (b), 30% slag/ binder mixtures (c) and 40% slag/ binder mixtures (d).

Comparing the results in Figures 5.19 (a-d), different silica fume forms have different mechanisms in the direct tensile performance of SFRGC mixtures. Incorporation of silica fume improves the load carrying capacity, although this effect is most marked

in samples with lower slag content (10% and 20%) (See Figure 5.19a and Figure 5.19b). This is due to replacement of relatively low reactivity material (fly ash) by highly pozzolanic material (silica fume) in the binder which accelerates the geopolymerization process, and enhances the bond between the matrix and the steel fibres, increasing the energy requirements for the fracture process. This improvement is less marked at higher slag contents, where increased slag amounts act to enhance the pozzolanic activity and reactivity, reducing the impact of silica fume addition. The influence of silica fume depends on particle size, and the finer silica fume (USF) is more capable of filling pores and producing a denser geopolymer matrix. However, the ultra-fine silica (SSF) has lower tensile strength than USF, possibly due to accelerated agglomeration of this very reactive material during the mechanical mixing of the initial material.

The inelastic behaviour of specimens reinforced with steel fibres could be observed as the first crack initiated from one side of the specimen and propagated slowly to the other side. After first cracking, the stress dropped, and the specimen changed stiffness. Some specimens experienced strain softening behaviour, due to some fibre pull-out from the weak strength geopolymer matrix. However, high slag content mixtures exhibited some ductility before tensile softening. The plain geopolymer specimens failed in a brittle manner and only a single failure crack in the middle portion was observed (Figure 5.20).



Figure 5.20: Failure mode of plain geopolymer mortar specimens (a) and SFRGC specimens (b).

5.3.3.3.3 Tensile bond characteristics of SFRGC with variant slag content and silica fume forms.

In the tensile stress-strain model, the main parameters are the tensile strength, the modulus of elasticity, fracture energy, and the shape of stress-strain curve. To examine the effect of geopolymer binder composition on the tensile performance of SFRGC specimens more fully, the experimental tensile response could be fitted quite accurately to a relatively bi-linear curve with initial cracking point and ultimate strength point. It has been indicated that the bi-linear relationship is a sufficient approximation to determine the most important tensile strength parameters for practical engineering applications (Cotterell and Mai, 1995), which reflect the mechanism of the interaction between the variant composition geopolymer matrix and the steel fibres (showing initial cracking point with matrix cracking, and maximum loading point with slip of fibres) (Figure 5.21).



Figure 5.21: Concept of bi-linear stress-strain curve of SFRGC.

The tensile response of SFRGC should exhibit linear elasticity up to the first cracking point, followed by nonlinear behaviour whereas strain hardening up to the maximum post cracking stress or softening material (Figure 5.21). Two stages of steel fibre response before destruction under tension load are defined, the first stage represents stress distribution during the elastic range before first cracking *E*, while the second stage corresponds to the post-cracking modulus E_{cr} . These parameters were calculated

from the stress-strain curves (Figure 5.18 and Figure 5.19) and the results are presented in Table 5.5, Figure 5.22 and Figure 5.23.



Figure 5.22: Tensile strength characteristics of binary blended SFRGC mixtures with variant slag content.






Figure 5.23: Tensile strength characteristics of ternary blended SFRGC mixtures with variant silica fume forms; 10% slag/binder mixtures (a), 20% slag/ binder mixtures (b), 30% slag/ binder mixtures (c) and 40% slag/ binder mixtures (d).

As can be seen from the results Figure 5.22, Figure 5.23 and Table 5.5, the Young's modulus of elasticity significantly changes with the binary and ternary binder composition of the geopolymer mixture. As slag content increased in the geopolymer mixture, the first cracking modulus of SFRGC mixtures considerably improved. The first cracking modulus of SFRGC mixtures increased by 120%, 300%, and 500% for the 20S-ST, 30S-ST, 40S-ST mixtures, respectively compared with the elastic modulus of the respective 10S-ST mixture (Table 5.5 and Figure 5.22). In addition, the results indicated that the post cracking modulus strength significantly increased with increasing slag content in the geopolymer mixture. The post cracking modulus increased from 140 MPa for the 10S-ST mixture to 200 MPa, 350 MPa, and 1000 MPa for the 20S-ST, 30S-ST, and 40S-ST mixtures, respectively (Table 5.5). This increment in the first cracking and post cracking modulus with slag content in the SFRGC mixtures lead to development of strain hardening behaviour under tension. This trend was confirmed as the post cracking tensile strain increased from 0.0013 to 0.0027 by increasing slag content in the geopolymer mixture from 10% to 40%. The increment in the first cracking and post cracking modulus is due to strength improvement in the geopolymer matrix with higher slag content, leading to a stronger interfacial bond between the steel fibres and the geopolymer matrix.

Inclusion of silica fume in the geopolymer matrix also effects the linear stage of the stress strain curve: the first cracking modulus of the 10S-ST mixture increased by 11%, 50% and 100% with utilization of DSF, USF and SSF, respectively (Figure 5.24). However, this trend is not apparent at higher slag contents as inclusion of SSF in the mixture reduced the elastic modulus, and USF incorporation tended to result in the most consistent improvement in elastic modulus values. The modulus of elasticity of the 30S-ST mixture changed from 8000 MPa to 8500 MPa, 10625 MPa, and 7500 MPa for ternary SFRGC mixtures containing DSF, USF, and SSF, respectively (Figure 5.24). Moreover, the post cracking strain capacity improved in the 30S-ST mixture from 0.0016 to 0.002, 0.0025, and 0.0012 for 10DSF, 10USF, and 5SSF, respectively (Table 5.5). The highest values of first and post cracking modulus of elasticity in SFRGC were found for the 40S-10USF-ST mixture, at 15400 MPa and 1125 MPa, respectively.

Based on the data listed in Table 5.5, the post cracking tensile strength increased with increasing slag content. Based on these results, the average tensile strength was increased by 100%, 250% and 340% for 20S-ST, 30S-ST and 40S-ST mixtures, respectively, compared with the respective value for the 10S-ST mixture. Combining slag with silica fume had differing effects on the tensile behaviour of SFRGC depending on the silica fume particle size distribution and slag content. The tensile strengths of 20% and 40% slag mixtures with 0%SF, 10%DSF, 10%USF and 5%SSF were 1.36 MPa, 1.46 MPa, 2.4 MPa, 1.6 MPa and 2.8 MPa, 2.8 MPa, 3.1 MPa, 2.3 MPa, respectively. Steel fibre reinforced geopolymer composites incorporating undensified silica fume show the highest post cracking tensile strength for all slag content mixtures.

Mix ID	First cracking strength	Post cracking strength	Tensile strain	First cracking	Post-cracking modulus <i>E</i> _{cr}
			capacity	modulus	
	[MPa]	[MPa]		<i>E</i> [MPa]	[MPa]
10S-ST	0.45	0.6	0.0013	2045	140
10S-10DSF-ST	0.8	0.96	0.0012	4000	160
10S-10USF-ST	1.6	2	0.0018	4000	290
10S-5SSF-ST	1.87	2.22	0.002	2600	185
20S-ST	0.75	1.36	0.002	4400	200
20S-10DSF-ST	1	1.46	0.0014	5500	300
20S-10USF-ST	1.75	2.4	0.002	8400	290
20S-5SSF-ST	1.26	1.6	0.0012	3400	340
30S-ST	1.6	2	0.0016	8000	350
30S-10DSF-ST	1.7	2.2	0.002	8500	275
30S-10USF-ST	1.7	2.6	0.0025	10625	385
30S-5SSF-ST	1.2	1.5	0.0012	7500	340
40S-ST	1.4	2.8	0.0027	11700	1000
40S-10DSF-ST	2	2.8	0.0027	11800	1025
40S-10USF-ST	2.3	3.1	0.0028	15400	1125
40S-5SSF-ST	1.65	2.3	0.0005	9100	4500



Figure 5.24: Modulus of elasticity of SFRGC with variant silica fume forms and slag content.

The energy absorption capacity represents the area under the stress-strain curve of the SFRGC up to 2% strain capacity after appearance of cracking (Figure 5.25). The results show that increasing the percentage of slag content in the geopolymer matrix generally increases the energy absorption capacity. The results also indicated that the inclusion of different silica fume forms influences the energy absorption capacity, with

the sample containing undensified silica fume giving the highest energy absorption, at 40% slag content. The results also show that inclusion of slurry silica fume in the 10% slag content mixture gives markedly superior energy absorption capacity than other forms of silica at this low slag content. This behaviour may be a result of the impact of fine-grained SSF on pozzolanic activity, enhancing the bond between the matrix and the steel fibres at lower slag contents. Despite this, Figure 5.22, Figure 5.23 and other tensile testing data (e.g. Figure 5.24, Figure 5.25 and related discussion) indicate that USF incorporation overall tends to produce materials with the most improved tensile performance.



Figure 5.25: Fracture energy of SFRGC with variant silica fume forms and slag content.

5.3.3.4 Relationship between compressive strength and direct tensile strength of SFRGC.

As geopolymer is a relatively new cementitious material, no equations have yet been established to define the relationships of mechanical properties of geopolymer material in structural concrete standards such as the European Standard (EN) 1992-1-1 (2004) and ACI Committee 318, (1999) (Neupane, 2016). This section is intended to examine the applicability of the existing relationship between the direct tensile strength and compressive strength of steel fibre reinforced geopolymer concrete. A series of regression analyses was undertaken to evaluate the relationship between direct tensile strength and compressive strength for binary and varying ternary SFRGC mixtures with curing time, and the results of these analyses are summarized in Table 5.6, Table 5.7 and Figure 5.26.



Figure 5.26: Relationship between compressive strength and tensile strength of SFRGC with variant silica fume and slag content; 0%SF (a), 10%USF (b), 5%SSF (c), and 10%DSF (d).

Table 5.6: Properties of regression lines of SFRGC mixtures at 10%, 20%, 30% and 40% slag to binder weight ratio.

Mixtures	Age	Regression	Slope	Intercept	coefficient of
		equations			correlation
(%)	[day]		[B]	[A]	[r]
Dinomonioso	3	$f_t = 0.130 f_c^{0.574}$	0.574	0.130	0.965
(EA/Slag)	7	$f_t = 0.041 f_c^{0.874}$	0.874	0.041	0.766
(I A Slug)	28	$f_t = 0.013 f_c^{1.345}$	1.345	0.013	0.957
Ternary mixes (10DSF)	7	$f_t = 0.016 f_c^{1.291}$	1.291	0.016	0.428
	28	$f_t = 0.199 f_c^{0.649}$	0.649	0.199	0.874
Ternary mixes (10USF)	3	$f_t = 0.167 f_c^{0.514}$	0.514	0.167	0.918
	7	$f_t = 0.039 f_c^{1.005}$	1.005	0.039	0.961
	28	$f_t = 0.536 f_c^{0.417}$	0.417	0.536	0.986
Ternary mixes (5SSF)	3	$f_t = 0.009 f_c^{1.584}$	1.584	0.009	0.978
	7	$f_t = 0.003 f_c^{1.748}$	1.748	0.003	0.869
	28	$f_t = 0.267 f_c^{0.5}$	0.500	0.267	0.224

Mix ID		Proposed	l	Experimental/ proposed ratio			
	3 days	7 days	28 days	3 days	7 days	28 days	
10S-ST	0.211	0.248	0.620	1.044	1.208	0.968	
10S-10DSF-ST	NA	NA	0.951	NA	NA	1.051	
10S-10USF-ST	0.338	0.436	1.967	1.064	0.940	1.017	
10S-5SSF-ST	0.036	NA	0.901	11.661	NA	2.463	
20S-ST	0.333	0.433	1.165	0.991	0.761	1.168	
20S-10DSF-ST	NA	0.539	1.850	NA	0.501	0.789	
20S-10USF-ST	0.608	0.865	2.459	0.971	1.086	0.976	
20S-5SSF-ST	0.243	1.283	1.510	1.031	0.398	1.059	
30S-ST	0.629	0.785	2.293	0.858	0.739	0.872	
30S-10DSF-ST	NA	0.553	1.962	NA	1.898	1.121	
30S-10USF-ST	0.709	1.190	2.622	0.846	1.118	0.992	
30S-5SSF-ST	0.607	1.080	1.803	0.840	0.750	0.776	
40S-ST	0.844	0.937	2.474	1.138	1.440	1.132	
40S-10DSF-ST	NA	1.481	2.585	NA	0.950	1.083	
40S-10USF-ST	0.959	1.617	3.036	1.147	0.884	1.021	
40S-5SSF-ST	1.239	1.426	1.907	1.049	1.122	1.206	

Table 5.7: Comparison of direct tensile strength (experimental and proposed) over curing time.

The non-linear relationship between the direct tensile strength and compressive strength of SFRGC is shown in Figure 5.26. The intercepts (A) and the slopes (B) represents the constant values in the regression equation $(f_t = A (F_c)^B)$ (Table 5.6). Each regression equation is applicable for all slag contents (10%, 20%, 30% and 40%) of experimental data over the curing age. Most of the regression equations gave a ratio closer to 1 even though it is slightly lower estimate the value for 28 days predicted equation containing DSF and SSF. Figure 5.26 and Table 5.7 show that the predicted tensile strength (f_t) at early and later age for binary and ternary blended SFRGC mixtures generally agreed with the test results. The coefficients of correlation of binary blended SFRGC mixtures were 0.965, 0.766 and 0.957 at 3 days, 7 days, and 28 days, respectively. The tensile strength of ternary mixture at 28 days strongly agreed with the compressive strength as the coefficients of correlation were 0.874 and 0.986 for 10%USF, and 10%DSF mixtures, respectively (Table 5.6). The existing equations did not give high accuracy however in the prediction of tensile strength (f_t) for ternary SFRGC containing SSF and lower slag content. This is also confirmed in Table 5.7: the experimental to the predicted tensile strength ratio was close to 1 for most of the binary and ternary mixtures apart from the ternary mixture containing 5%SSF. The predicted tensile strength of ternary SFRGC mixtures containing USF gave the best fit to the test results at all examined ages, compared with ternary SFRGC specimens containing DSF and SSF. Based on the test results, the regression equation to predict

tensile strength from the compressive strength of ternary mixtures containing USF, for 3 days curing, was found to be $f_t = 0.167 f_c^{0.514}$ and the same relationship for 28 days was found to be $f_t = 0.536 f_c^{0.417}$.

The empirical relation between the mean tensile strength and the compressive strength of the OPC concrete suggested by ACI-363R, (1992) and Neville, (1995) are expressed as follows respectively;

$$f_t = 0.59 f_c^{0.5} \quad [\text{MPa}] \tag{5-1}$$

$$f_{\rm f} = 0.23 f_{\rm c}^{0.67}$$
 [MPa] (2-5)

Some studies have proposed different models for the experimental relationship between compressive strength and splitting tensile strength for geopolymer materials. As the strength of the final products are influenced by various parameters such as type of source material and concentration and type of alkaline activators used (Duxson, 2007; Joseph and Mathew, 2012; Ryu *et al.*, 2013) there is a variation in results:

$$ft = 0.59 f_c^{0.5}$$
 [MPa] (Sofi *et al.*, 2007) (5-3)
$$ft = 0.616 f_c^{0.5}$$
 [MPa] (Tempest, 2010) (5-4)

The measured 28 days direct tensile strengths and the equations derived in this thesis were compared with the predicted tensile strength of the OPC concrete according to the ACI 363 code, and the equations of Neville, (1995) and Sofi *et al.*, (2007). The results of experimental and predicted tensile strength from the predicted equations in ACI Committee 318, (1999), Neville, (1995), and the current study are listed in Table 5.8 and Figure 5.27. The average experimental to predicted tensile strength ratio of binary and ternary blended SFRGC mixtures is also calculated and presented.

Mix ID	Experi	mental days]	Predicted tensile strength			Experimental/predicted ratio				
	Tensile strength	Comp. strength	ACI 363	Neville	Proposed	Sofi	ACI 363	Neville	Proposed	Sofi
10S-ST	0.60	17.70	2.48	1.58	0.62	2.10	0.24	0.38	0.97	0.29
20S-ST	1.36	28.28	3.14	2.16	1.16	2.66	0.43	0.63	1.17	0.51
30S-ST	2.00	46.80	4.04	3.03	2.29	3.42	0.50	0.66	0.87	0.58
40S-ST	2.80	49.52	4.15	3.14	2.47	3.52	0.67	0.89	1.13	0.80
10S-10USF-ST	2.00	22.60	2.80	1.86	1.97	2.38	0.71	1.08	1.02	0.84
20S-10USF-ST	2.40	38.60	3.67	2.66	2.46	3.11	0.65	0.90	0.98	0.77
30S-10USF-ST	2.60	45.00	3.96	2.95	2.62	3.35	0.66	0.88	0.99	0.78
40S-10USF-ST	3.10	64.00	4.72	3.73	3.04	4.00	0.66	0.83	1.02	0.78
10S-5SSF-2ST	2.22	11.40	1.99	1.17	0.90	1.69	1.11	1.89	2.46	1.32
20S-5SSF-2ST	1.60	32.00	3.34	2.35	1.51	2.83	0.48	0.68	1.06	0.57
30S-5SSF-2ST	1.40	45.60	3.98	2.97	1.80	3.38	0.35	0.47	0.78	0.41
40S-5SSF-2ST	2.30	51.00	4.21	3.20	1.91	3.57	0.55	0.72	1.21	0.64
10S-10DSF-2ST	1.00	11.14	1.97	1.16	0.95	1.67	0.51	0.86	1.05	0.60
20S-10DSF-2ST	1.46	31.04	3.29	2.30	1.85	2.79	0.44	0.64	0.79	0.52
30S-10DSF-2ST	2.20	34.00	3.44	2.44	1.96	2.92	0.64	0.90	1.12	0.75
40S-10DSF-2ST	2.80	52.00	4.25	3.25	2.59	3.61	0.66	0.86	1.08	0.78

Table 5.8: Comparison of 28 days tensile strength (experimental and theoretical).



Figure 5.27: Comparison the relationship between compressive strength and tensile strength of SFRGC according to ACI318, Neville, proposed and experimental; 0%SF (a), 10%USF (b), 5%SSF (c), and 10%DSF (d).

As can be observed from the results listed in Table 5.8 and Figure 5.27, most of the predicted tensile strengths according to ACI-363R, (1992) Building Code significantly underestimate the experimental tensile strength results. However, the equation of Neville, (1995) gave a ratio closer to 1, even though it slightly underestimated the experimental value for binary blended SFRGC containing low slag content (10%-30%) and ternary blended geopolymer mixture containing SSF. For ternary blended SFRGC mixtures containing USF, the empirical expression proposed in this study and that proposed by Neville, 1995 gave a closer value to the experimental results. Considering previous models and experimental results, the 28 days proposed relationship of tensile strength against compressive strength for binary mixtures was found to be $f_t = 0.013 f_c^{1.345}$, and the same relationship found to be $f_t = 0.199 f_c^{0.649}$, $f_t = 0.536 f_c^{0.417}$, and $f_t = 0.267 f_c^{0.5}$, for ternary mixes containing DSF, USF, and SSF, respectively. It should be noted however that further research on the mechanical properties of SFRGC is required to propose a general relationship between the compressive strength and the tensile strength, as only a small number of specimens were used in the present study.

5.3.4 Fourier Transform Infrared Spectroscopy (FTIR) Analysis.

Structural changes in the geopolymer mixture with different mix compositions were investigated using Fourier transform infrared (FTIR) spectroscopy over a period of time up to 56 days. In order to compare the effect of using different silica fume forms in the geopolymer mixture at different slag contents, eight geopolymer paste mixtures containing 0%SF, 10USF, 10DSF, and 5SSF at 10% and 40% slag to binder wt. ratio were analysed in this study. FTIR spectra of the binary and ternary blended geopolymer mixtures with different slag content and silica fume forms are given in Figure 5.28.





Figure 5.28: IR spectra of geopolymer pastes with different mix designs at 7 days (a), 28 days (b), and 56 days (c).

The FTIR spectra of the ternary geopolymer paste samples containing silica fume show some differences when compared to the spectra of the binary blended geopolymer paste samples. Figure 5.28 (a) shows the FTIR spectra of geopolymer paste mixtures containing 10% slag to binder wt. ratios at 7 days. The broad bands appearing at 3000-3500 cm⁻¹ and 1630-1645 cm⁻¹ were due to O-H stretching and bending vibrations of HOH, respectively (Alvarez-Ayuso et al., 2008). The existence of these bonds was detected in all geopolymer paste mixtures, which is related to entrap water molecules in the geopolymer network and indicated the geopolymerization reaction of alkaline solution into geopolymer paste. These bands are found at 3352, 3352, 3348 and 3350 cm⁻¹ for 10S, 10S-DSF, 10S-10USF, and 10S-5SSF geopolymer pastes, respectively. The bands seen at 1390-1410 correspond to the stretching vibration of O-C-O, which was attributed to the atmospheric carbonation reaction in the geopolymer specimens (Zaharaki et al., 2010). The strong band peaks at around 950-1000 cm⁻¹ are related to Si-O and Al-O asymmetric stretching vibrations, and are the fingerprint of the geopolymerization process (Phair and Van Deventer, 2002). The peaks in ternary geopolymer containing silica fume were shifted to a higher frequency at 959-973 cm⁻¹ compared to 958 cm⁻¹ for the binary fly ash and slag based geopolymer paste. The shift in the position of this broad band depends on the aluminium to silicate ratio, and this variation in the band frequency indicates a chemical change in the geopolymer structure when utilizing different silica fume forms. The 10S-10USF geopolymer paste showed the largest magnitude of shift to around 972 cm⁻¹, indicating more polymerised units of Si-O were established and added more strength to the mixtures (Lecomte *et al.*, 2006).

At higher slag content, Figure 5.28b shows similar observations to 10% slag to binder wt. ratio mixtures as the peak bands ranging from 950-1000 cm⁻¹ were also clearly presented. Si-O and Al-O symmetric stretching vibrations of the 40S geopolymer paste were shifted to the lower frequency peak at around 947 cm⁻¹ which was influenced by presence of (Ca) in the slag. The inclusion of a higher slag content in the geopolymer also promoted an increase in the asymmetry of Si-O and Al-O stretching vibrations of SiO₄ and AlO₄ of geopolymeric gel and mixed C-A-S-H gel of geopolymer cement (described in detail in Chapter 4). Inclusion of silica fume shifted the banding to 951 cm⁻¹, 954 cm⁻¹, and 956 cm⁻¹ for 40S-5SSF, 40S-10USF, and 40S-10DSF geopolymer paste, respectively. These results support the fact that the particle size of the examined material considerably influences the infrared intensity. The small size of the silica particles and their large surface to volume ratios cause the FTIR frequencies to differ greatly from those at larger silica particle size (Ernst et al. 2007).

The spectra of binary and ternary blended geopolymer materials at the ages of 28 days and 56 days are given in Figure 5.26 (c-f). The same trend is indicated at 56 days, the stretching vibration of the Si-O and Al-O bending modes were 955, 960, 963 and 955 for 40S-5SSF, 40S-10DSF, 40S-10USF, 40S mixtures, respectively. The bending vibrations were 971, 975, and 963 for 10S-5SSF, 10S-10DSF, 10S mixtures, respectively. These changes illustrate strengthening of the structure of the geopolymer, and influence the strength development behaviour under room temperature. The curing time could be attributed to a progressive increase in the overall degree of crosslinking of the gel over time, potentially through the initial formation of a C–S–H type gel followed by more gradual development of an N–A–S–H type gel with a higher crosslinking degree. The final position of these bands (40% slag mixtures) are at a slightly lower wavenumber than in the system containing low slag content (10% slag mixtures), indicating that the presence of high calcium content does lead to some differences in the gel network structure at later age (Nath and Kumar, 2013; Ismail *et al.*, 2014).

5.3.5 Scanning Electron Microscope (SEM) Analysis.

Scanning electronic microscopy imaging was carried out to examine the microscopic characteristics of the geopolymer binder materials, and the effect of slag content and silica fume forms on the microstructure of the geopolymer samples. Primary material particles (i.e. fly ash, silica fume and slag) and plain geopolymer and SFRGC samples (taken post-failure from 28 day cured samples used in tensile strength testing) were carbon sputter coated and then imaged using a Leo S420 stereoscan SEM with an accelerating voltage range of 1-30 kV. SEM imaging of the primary materials (Figure 5.29) showed that fly ash and silica fume particles generally consist of spherical and near-spherical primary particles (Figure 5.29a and Figure 5.29c); larger agglomerates of silica fume particles are formed in densified silica fume (Figure 5.29d) while slag (Figure 5.29b) consists dominantly of mixed size angular particles.



Figure 5.29: SEM images of the primary materials, fly ash (a), Slag (b), USF (c), and DSF (d).

5.3.5.1 Microstructure of ternary blended plain geopolymer mortar.

In order to compare the microstructures of geopolymer mortars with variant silica fume forms and to evaluate the effect of slag content on the ternary geopolymer microstructure, eight geopolymer mortars were prepared by replacing fly ash with 10% DSF, 10% USF and 5% SSF at 10% and 40% slag replacement content. The effect of curing time at early age (7 days) and later age (28 days) on the microstructure of geopolymer mortar is also examined (Table 5.9 and Table 5.10).

Table 5.9: SEM images of 10% slag replacement geopolymer mortar, after 7 days and 28 days ambient temperature curing, containing varying SF forms.

Mix ID	At 7 days	At 28 days
[A] 10S	Mag = 10 00 K.X. Mar Becktor # SEF EHT = 2000 M.X Mar Becktor # SEF	Mg = 10.00 K Year Dete day = 81 EHT = 20.00 k/ Year Dete day = 81
[B] 10S- 10DSF	Mag = 10.00 K X Para Detastor = 9.51 ENT = 20.00 K/ Para Detastor = 9.51	Mgg = 10.00 KX 10m² EHT + 22.00 M² 10m²
[C] 10 S- 10USF	Mage 10.00 KT Par Exect = SE1 D11 - 2003W Par Exect = SE1	Mg = 10.00 KX Tam Detector = SE1 EHT = 20.00 KV Tam Detector = SE1



Table 5.10: SEM images of 40% slag replacement geopolymer mortar, after 7 days and 28 days ambient temperature curing, containing varying SF forms.





Table 5.9a and Table 5.10a shows the area on a surface of 10S and 40S mixtures at x10000 magnification. The micrographs of the 10S mixture show a high proportion of partially reacted fly ash spheres, and agglomerated slag particles remaining in the matrix. This is due to the low pozzolanic reactivity of fly ash with low slag content cured under ambient temperature. However, the geopolymer sample with high slag content (40S mixture) (Table 5.10a) shows different microstructures, which form a denser matrix than the 10S mixture. Glassy crusts covering fly ash particles can be observed, as a result of reactions on the surface of the particles. Increasing the amount of GGBS significantly increased the calcium content in the mixture, leading to the formation of a calcium alumino-silicate hydrate (C–A–S–H) gel. Therefore, inclusion of GGBS introduced additional calcium bearing compounds and contributed to additional binding products, which improve the compressive strength, reduce porosity and modify the setting behaviour of geopolymeric gels at early ages. This enables the formation of a more compact gel structure and consequently improves mechanical properties.

Inclusion of silica fume had varying effects on sample microstructure. The texture of the hydration products of the 10% slag to binder geopolymer mortar with DSF was visibly different from samples with USF and SSF. For 10%DSF (Table 5.9) the observed SEM image shows no significant difference from the control mix (Table 5.9a), as the large densified silica fume particles cause lower packing and lower the pozzolanic activity of the silica fume. However, the micrographs show that the geopolymerization products of the mixtures containing smaller particle sizes of silica fume (for undensified ($37\mu m$) and slurry silica (200nm)) consisted of well-connected structures, and compacted formations of hydration products were observed. In addition

to acting as a physical filler in the matrix structure, silica fume acts as a source of high (85%-95%) reactive silica content leading to formation of more calcium aluminosilicate hydrate (C-S-A-H) gels which co-exist with sodium alumina-silicate hydrate (N-A-S-H) (Siddique, 2011). This is also indicated by quantitative porosity results, as total porosity of the 10% slag replacement mixture was considerably reduced from 30 to 23-25% by inclusion of USF and SSF. These reductions in total porosity indicate an increase in the matrix density, and also improve the compressive strength of the geopolymer mortar. At higher slag content mixtures (Table 5.10), it can be seen that inclusion of different silica fume forms had similar effects however, all the examined mixtures had a dense and compact structure due to extra precipitation of (C-N-A-S-H) from calcium minerals introduced in the slag. Moreover, a considerable increase in compressive strength was also obtained when the slag content replacement increased. It is also noted that increasing the curing time from 7 days to 28 days significant increases microstructural development of geopolymer mortar cured under ambient temperature. For all 10% slag to binder weight mixtures, increasing curing time lead to compaction of the geopolymer microstructure. However, these difference in SEM images was less pronounced at higher slag content mixture with increasing curing time. This can be explained the role of slag content on the microstructure and strength development of geopolymer mortar at early age.

5.3.5.2 Microstructure of SFRGC mixture.

Scanning electron microscope (SEM) imaging of the steel fibre-geopolymer matrix interface and fibre surface texture was also conducted in order to assess the effect of the microstructural characteristics on mechanical performance in SFRGC. SEM images of steel fibre reinforced geopolymer for 10S-10USF-2ST, 40S-10DSF-2ST and 40S-10USF-2ST mixtures are shown in Table 5.11. After the end of the tensile tests, samples were collected from parts of the specimens near the cracks failure to examine sample microstructure after 28 days.

Table 5.11: SEM micrographs of steel fibre reinforced geopolymer composite, imaged perpendicular to the fracture surface following failure during tensile tests.



The 28-day age microstructures of SFRGC mixtures were used to compare and analyse the differences introduced by changes in slag content and silica fume forms in the geopolymer. As can be observed from Table 5.11, the steel fibre surface is considerably effected by the geopolymer matrix composition. Increasing the slag content and inclusion of silica fume leads to enhanced interfacial properties. A relatively smooth steel fibre surface is seen in the geopolymer mixture containing 10% slag content, conversely the high slag (40%) content samples show the steel fibre surface covered with geopolymer matrix (Table 5.11b). In addition, the inclusion of different forms of silica fume in the geopolymer composite effects the steel fibrematrix contact, which is evidenced by the presence of more hydration production on the steel surface in the case of USF geopolymer mixtures (Table 5.11c). These hydration products create a stronger bond at the interface between the matrix and the steel fibres, and resist pull-out failure of the examined specimens which leads to an increase in the ultimate load and increasing sample ductility by improving the carrying capacity in the post cracking stage. These enhanced interfacial properties have a direct effect on the tensile strength characteristics, in agreement with the mechanical testing results presented in Section 5.3.3.3.

5.3.6 Concluding remarks.

Novel cement-free geopolymer composites, reinforced with steel fibres and cured under ambient temperatures, have been developed in this study. The present study investigated the fresh, hardened and microstructural properties of plain geopolymer mortar and SFRGC. Thirty-two geopolymer mixtures were used to examine the effect of (a) varying slag contents, and (b) varying silica fume forms on engineering performance. The following conclusions can be drawn from the results presented in this Chapter:

5.3.6.1 Fresh geopolymer mortar characteristics.

- Increasing the slag content in the fly ash and slag based geopolymer mortar decreases the workability and accelerates the setting times (initial and final) and mortar hardening.
- 2. The inclusion of silica fume in the geopolymer mortar has various effects on the flow characteristics of fly ash and slag based geopolymer mortar. In the case of undensified and slurry silica, the workability and setting time were considerably reduced. This is attributed to the instantaneous interactions between the very fine silica particles and the alkaline solution, and the formation of a gel characterised by high water retention capacities. The addition of densified silica fume did not significantly affect workability.

5.3.6.2 Hardened geopolymer mortar and SFRGC characteristics.

1. Compressive strength of plain geopolymer and SFRGC was increased as the slag content was increased and with the age of the specimens.

- Ternary geopolymer mixtures based on combined use of silica fume, slag and fly ash show a notable improvement in the rate of strength development of plain and SFRGC over control binary mixtures containing GGBS and FA.
- 3. Utilization of USF and SSF considerably improved the compressive strength of plain geopolymer and SFRGC. However, DSF showed less effect or lower compressive strength than control binary GGBS and FA mixtures. Moreover, these effects are more pronounced at lower slag content rather than at higher slag content.
- 4. The Young's modulus and ultimate tensile strength increased with increasing slag content in the SFRGC mixtures. Moreover, inclusion of USF and SSF improved the tensile strength of SFRGC. The 40S-USF mixture showed the highest tensile strength value of around 3.1 MPa.
- 5. Post cracking behaviour and energy absorption capacity considerably improved by increasing slag content and including fine particle sizes of silica fume (USF).
- 6. Overall, increasing the curing time considerably improved the compressive, tensile and post cracking behaviour of SFRGC cured under ambient temperature. The 28 days proposed relationship of tensile strength against compressive strength for binary mixtures was found to be $f_t = 0.013 f_c^{1.345}$, and the same relationship found to be $f_t = 0.199 f_c^{0.649}$, $f_t = 0.536 f_c^{0.417}$ and $f_t = 0.267 f_c^{0.5}$ for ternary mixes containing DSF, USF, and SSF, respectively.

5.3.6.3 Microstructural properties.

- Microstructural observation by SEM, and porosity results, confirm that the incorporation of slag and silica fume as a partial fly ash replacement in geopolymer mortars densified the microstructure, leading to an improvement in mechanical strength.
- 2. A relatively good bond between the matrix and the steel fibres was also evidenced by the presence of geopolymer hydration products on the surface of the steel fibres in specimens with high slag content and USF.

The findings of the current research show that binary geopolymer mixes (FA and GGBS) enhanced both mechanical strength and microstructure. Moreover, higher strength with a compacted microstructure was found when utilizing silica fume, even

without any heat curing treatment, which makes the proposed method suitable for in situ applications. Further studies should investigate the effect of silica fume forms on the rate of strength development at late states of the geopolymerization process. Also, superior durability characteristics of fibre reinforced geopolymer concrete against chloride resistance could be achieved by using ultra fine silica fume. It is noted that various fibre types could be utilized to improve the strain hardening cementitious material (Choi *et al.*, 2012; Shaikh, 2013a). However, due to the large number of geopolymer matrix composition mixtures listed in this study, the discussion here is limited to a 2%volume fraction of steel fibre. Further investigation on developing sustainable strain hardening geopolymer concrete by using various fibres types and contents such as glass fibre, and Polyvinyl Alcohol (PVA) fibre, would be useful. Aspects of this are explored in Chapter 6.

CHAPTER 6:

DEVELOPMENT OF STRAIN HARDENING CHARACTERSTICS OF FIBRE REINFORCED GEOPOLYMER CONCRETE CURED UNDER AMBIENT TEMPERATURE

6.1 Introduction.

Strain hardening cementitious concrete (SHCC) is a type of fibre reinforced concrete with enhanced mechanical properties, including strain hardening and ductility. However, a high cement content is commonly used in the SHCC mixture design, which means that its production and use is more energy intensive than conventional concrete. Using high cement amounts leads to increased heat of hydration, higher shrinkage and is more energy intensive (Altwair et al., 2012). In addition, use of a high ordinary Portland cement (OPC) content has negative environmental impacts associated with the cement manufacturing process - production of 1 ton of cement generates 1 ton of greenhouse gas emissions (Gartner, 2004; Turner and Collins, 2013). Partial replacement of OPC by waste materials such as fly ash (FA) has been reported in some studies as a possible solution to this problem (Nematollahi et al., 2015). Bernal et al., (2012) examined the feasibility of creating an engineering cementitious composite, taking into account environmental sustainability considerations, by using high FA content (up to 85% by weight) cement. Their results showed that a high volume of FA amount can reduce the drying shrinkage, crack width, and improve tensile ductility, although this also reduced the 28 days compressive strength. Choi et al., (2012) investigated the effect of partial replacement of cement by recycled materials on the mechanical properties of strain hardening cementitious materials (SHCC). Alternative by-product materials (FA, sand, and polyethylene terephthalate (PET) fibers) were used to partially replace cement, silica sand, and polyvinyl alcohol (PVA) fibers, respectively. Their results indicated that FA improves both bending and tensile behavior due to generation of stronger bonding between the PVA fibres and the cement matrix. The SHCC containing PET fibres however performed relatively poorly in tensile and bending strength tests.

Geopolymer (cement-free) represents the most promising sustainable material alternative to ordinary Portland cement. As discussed in previous chapters, heat treatment however is crucial when using this material, to provide comparable mechanical properties to conventional concrete, and there are a number of practical limitations in the application of heat curing in large-scale structures (Al-Majidi *et al.*, 2016b). This heat treatment leads to increased cost, effects the material sustainability, and generates practical production and handling issues, preventing in situ application of geopolymer concrete at large scales (Lee and Lee, 2013; Deb *et al.*, 2014; Nath and Sarker, 2014).

The mechanical performance of fibre reinforced cementitious composites depends on the material parameters (both of the fibres themselves, and the cementitious matrix). These parameters include strength, aspect ratio, Poisson's ratio and shape of fibres; the stiffness, strength, and shrinkage of the matrix; and, frictional bond properties and the physio-chemical interaction at the interface between the cementitious matrix and the fibres. Although geopolymer concretes are a growing area of research and development, there are as yet only a small number of studies examining fibre reinforced geopolymer composites in the open literature compared with traditional fibre reinforced cement concretes (Duran Atiş et al., 2009; Bernal et al., 2010; Natali et al., 2011). In the previous chapters (Chapter 4 and Chapter 5), the development of more user-friendly geopolymers has been proposed in order to enhance the strength of the geopolymer material as well as reduce costs and energy consumption, and promote easier handling application (Davidovits, 2011; Al-Majidi et al., 2016b). The geopolymer matrix was produced using ternary geopolymer binder (FA, GGBS and silica fume) mixed with a low content and concentration of potassium silicate alkaline activator. There are no published studies to date, however, on user friendly geopolymer systems with strain hardening characteristics.

This chapter focuses on the development and evaluation of the mechanical properties of a novel, effective and sustainable strain hardening fibre-reinforced geopolymer composite material, cured under ambient temperature and thus suitable for cast-inplace application (addressing thesis objective 2). In particular, the effect of incorporation of discontinuous fibres on the mechanical performance and on the microstructure of the composite geopolymer materials have been evaluated via compressive, flexural and direct tensile tests, and scanning electron microscopy. A range of different fibre types, aspect ratios, and volume fractions have been examined.

6.2 Experimental procedures.

Fourteen different mixtures were prepared, to evaluate the effect of incorporation of variant volume fractions and aspect ratios of discontinuous steel (ST), polyvinyl alcohol (PVA), and glass fibres on the mechanical performance and microstructure of the composite (Table 6.1). A Zyklos high shear mixer (Pan Mixer ZZ 75 HE) was used to manufacture the fibre-reinforced geopolymer composite. The binder powder materials (FA, GGBS, and SF) were dry mixed for 5 min and then the liquid phase was added and the mixer run for another 5 minutes. After that, steel fibres were gradually added after sieving through an appropriate steel mesh at the top of the mixer, in order to ensure uniform fibre dispersion in the geopolymer mix. Finally, sand was added to the mixer, and the mixer was run for another 3 minutes to give a total mixing time of 13 minutes. After demoulding, the moulds were covered with plastic sheets to prevent moisture loss and cured at room (i.e. ambient) temperature up to the testing date. The mechanical properties and microstructural characteristics of the composites were examined via compressive, flexural and direct tensile tests, and scanning electron microscopy. Flexural load-deflection curves have also been used to determine flexural strength, residual strength and flexural toughness. The strength development of FRGC over the curing time was also examined.

An identification nomenclature was given to each sample series in (Table 6.1) where: the first letter denotes the volume fraction of fibre (1%, 2%, and 3%); the second letter denotes the type of fibre (ST= steel, PVA= polyvinyl alcoholic, HE= hooked end); and the third letter denotes the length of fibre (that is, for the straight steel fibres, 6 mm and 13 mm). For example, specimen 1HE-1ST13 is a specimen cast using geopolymer has a 1% volume fraction of hook end fibre with 1% volume fraction of straight steel fibre with 13 mm.

MIX ID	OPC	FA/	GGBS /	SF/	Sand	K2SiO2/	Water/	Fibre
	$[Kg/m^3]$	Binder	Binder	Binder	[Kg/m ³]	Binder	Binder	V _f [%]
OPC mortar	650	-	-	-	1525	-	0.35	0
GP mortar	_	0.5	0.4	0.1	1052	0.12	0.25	0
2ST6	-	0.5	0.4	0.1	1052	0.12	0.25	2
3ST6	_	0.5	0.4	0.1	1052	0.12	0.25	3
1ST13	_	0.5	0.4	0.1	1052	0.12	0.25	1
2ST13	_	0.5	0.4	0.1	1052	0.12	0.25	2
3ST13	_	0.5	0.4	0.1	1052	0.12	0.25	3
3[ST6-ST13]	-	0.5	0.4	0.1	1052	0.12	0.25	3
1HE	-	0.5	0.4	0.1	1052	0.12	0.25	1
1HE-1ST13	-	0.5	0.4	0.1	1052	0.12	0.25	2
1HE-2ST13	-	0.5	0.4	0.1	1052	0.12	0.25	3
1PVA	-	0.5	0.4	0.1	1052	0.12	0.25	1
2PVA	-	0.5	0.4	0.1	1052	0.12	0.25	2
1Glass	-	0.5	0.4	0.1	1052	0.12	0.25	1

Table 6.1: Mixture compositions of fibre reinforced geopolymer concrete.

6.3 Results and Analysis.

6.3.1 Compressive strength Testing.

6.3.1.1 Effect of curing time on the strength development of FRGC.

Examination of the compressive strength development of strain hardening geopolymer concrete over curing time (3, 7 and 28 days) was conducted by using nine cubes with 50 mm sides for each mixture (Figures 6.1 and 6.2), using a Denison Avery 2000 KN test machine with loading rate of 45 kN per minute (ASTM C109/109M, 2007). This cubic size was valid only for micro fibre mixtures as the macro fibre length exceeded the limit of the cube size. Each data point corresponds to an average of three specimens.



Figure 6.1: Influence of steel fibre aspect ratio and volume fraction on the compressive strength development of FRGC over curing time (50 mm cubic specimens).



Figure 6.2: Influence of PVA and glass fibre on the compressive strength development of FRGC over curing time (50 mm cubic specimens).

The compressive strength of SFRGC increased with increasing curing time for all the examined mixtures (Figure 6.1). At early age (3 days), the inclusion of steel fibre reduced the compressive strength for all steel fibre reinforced mixtures apart from the 3ST13 and 3[ST6-ST13] mixtures. The compressive strength reduced by 18%, 45%, 35%, and 16% for 2ST6, 3ST6, 1ST13, 2ST13 mixes respectively, compared with the control geopolymer mortar without steel fibre. With increasing curing time from 3 days to 7 days, the compressive strength of steel fibre considerably improved. The compressive strength was increased by 60%, 190%, 155%, 55%, 65%, and 30% for 2ST6, 3ST6, 1ST13, 2ST13, 3ST13 and 3[ST6-ST13] mixtures, respectively, compared with the respective compressive strength values at 3 days. This observation indicates similar behaviour to that reported in conventional ordinary Portland concrete, which undergoes a hydration process and develops strength over time. Inclusion of PVA and glass fibre reduced the early age compressive strength compared both with the plain geopolymer mortar and SFRGC mixtures at the same volume fraction (Figure 6.2). The maximum compressive strengths were achieved at 28 days, at ca. 45 MPa, 59 MPa, 57 MPa, 45 MPa, and 42 MPa for PG, 2ST6, 2ST13, 2PVA and 1Glass mixtures, respectively. This overall trend in increasing compressive strength over time is due to the low strength of the geopolymer matrix at early ages, which weakens the interfacial bonds between the fibre and the geopolymer matrix. At later ages the compressive strength of FRGC significantly improves due to infilling of the porous microstructure of the geopolymer matrix by formation of more hydration products, leading to improvement in the fibre–matrix bond.

6.3.1.2 Effect of fibre type on the compressive strength of FRGC.

From the above observations, the optimum compressive strength of all the examined mixtures at 28 days is compared in Figure 6.3. Each column corresponds to an average of three cubic specimens with 100 mm sides while the scatter of the experimental results is also presented.



Figure 6.3: 28d-Compressive strength of plain and fibre reinforced geopolymer composites (100 mm cube specimens).

The compressive strength value of the plain geopolymer mortar without fibres was 43.7 MPa. The incorporation of steel fibres increases the compressive strength by about 15-25 MPa depending on the fibre aspect ratio, shape and dosage rates. The resistant capacity of the ST13 mixture under compression increased by increasing the fibre dosage rates, although this trend was not apparent for the ST6 mixtures, where the compressive strength decreased with increasing fibre content. This is due to the high number of short steel fibres at high volume fraction (V_f) (3%) (*i.e.* double that of the ST13 fibre at the same V_f), leading to redistribution of the voids structure, and weakening of the interfacial bonding between the fibre and geopolymer binder grains under compression. The 3ST13 mixture showed the highest strength value, of around 70 MPa. The increase in compressive strength for the geopolymer with macro hooked

end fibres was ca. 13 MPa. The hybrid macro and micro steel fibre (ST13) also showed improvement in compressive strength, depending on the dosage of straight ST13 fibres. The compressive strength of HE1050 fibres with a straight aspect improved the compressive strength to 62 MPa and 67 MPa for 1HE-1ST13 and 1HE-2ST13 specimens. This increment in the compressive strength is due to the ability of steel fibre to restrain the extension of cracks, reduce the stress concentration at the tip of cracks, and change the direction of cracks leading to a delay in their growth rate (Afroughsabet and Ozbakkaloglu, 2015). Reinforcing with glass and PVA fibres however did not significantly alter the compressive strength value compared to ordinary Portland cement and the geopolymer control sample. The compressive strength of 1PVA, 2PVA and 1Glass mixtures was 39 MPa, 42 MPa and 34 MPa.

6.3.2 Flexural strength testing.

6.3.2.1 Parameters describing the deflection hardening behaviour.

• Flexural specifications boundary of Fibre reinforced concrete (FRC).

The flexural performance of FRC can be classified as either deflection softening or hardening behaviour, based on the change of load carrying capacity after first cracking (Kim *et al.*, 2011; Shaikh, 2013a). According to ASTM C1609 / C1609M-05, (2005) and ASTM C1018-97, (1997); (Haider, 2014), the first cracking of SHGC is determined as the point where nonlinearity in the load–deflection relationship becomes evident. The load initiating first cracking in the specimens is termed the first peak load (P_1), while the second peak load represent the greatest value of the load on the load deflection curve of FRC. Fibre reinforced concrete exhibiting deflection hardening behaviour shows higher load carrying capacity after first cracking compared with deflection softening FRC (Figure 6.4).



Figure 6.4: Typical load–deflection curve for fibre reinforced concrete and flexural performance evaluation based on ASTM C1609-05.

The first cracking and ultimate flexural strength for each material can be determined by testing standard prism specimens ($100 \times 100 \times 500$ mm) at 28 days and using the following equation.

$$f_{1,2} = \frac{P_{1,2}L}{bd^2} \tag{6-1}$$

Where; Span length (*L*) equal to 450 mm (the distance between the supporting rollers), and b = 100 mm and d = 100 mm are the width and the height of the composite panels, respectively. The flexural test set-up details used here are presented in Figure 6.5. The testing machine was operated in a 'closed loop' to load the prisms at a fixed deflection rate of 0.24 mm/min. Two Linear Variable Displacement Transducers (LVDTs) were attached to a yoke frame which was used in order to eliminate any induced displacements at the supports during that loading.



Figure 6.5: Bending specimen test set-up.

• Concept of flexural toughness and residual strength.

In general, toughness is defined as a measure of energy absorption capacity, and is used to characterize fibre reinforced concrete's ability to resist fracture when subjected to flexural loads. Engineering toughness measures for FRC are usually obtained from the load-deflection of FRC beams subjected to four-point (or three-point) loading. The Japanese standard (JSCE-SF4, 1984) defines toughness using the load deflection curve of FRC until specific deflection (1/150 times the span) (Figure 6.6). The flexural toughness is dependent on the dimensions of the specimen since these will influence the total flexural load it can carry (Le, 2008). The ASTM C 1018-97 (1997) standard suggests non-dimensional performance (toughness indices) should be obtained by dividing the area up to a prescribed multiple of the first-crack deflection by the area until the first-crack deflection. It estimates flexural toughness indices I₅, I₁₀ and I₂₀ at four specific deflections (δ_1 , $3\delta_1$, $5.5\delta_1$ and $10.5\delta_1$) to describe whether pre-cracking or post-cracking strength is greater. The δ_1 represents the deflection at which the first crack is deemed to have occurred, as presented in Figure 6.7. The ACI Committee 544, (1988) recommendation defines the toughness index (I_t) that is the difference between the areas under the entire load-deflection curves of the fibre reinforced beam and load-deflection curve of an identical specimen without fibres. Thus, It is a measure of the improvement in toughness relative to the unreinforced matrix. This method is presented in Figure 6.8.



Figure 6.6: JCI SF4 standard measures of FRC toughness characterization.



Figure 6.7: ASTM C1018 standard measures of FRC fracture toughness indices.



Figure 6.8: ACI Committee 544 Measures of FRC toughness Characterization.

The ASTM C1609 / C1609M-05, (2005) standard defines the residual load values $(P_{600}^{\ D} \text{ and } P_{150}^{\ D})$ and corresponding residual strength $(f_{600}^{\ D} \text{ and } f_{150}^{\ D})$ which is determined at a net deflections of L/600 and L/150 for a beam with a depth of D. Toughness T_{600} , and T_{150} were identified which represent the area under the load-deflection curve up to the deflection points *L/600*, and *L/150*, respectively (Figure 6.9), where the *L/600*, and *L/150* deflection correspond to 0.75 mm, and 3 mm, respectively, for the specimen clear span of 450 mm.

$$f_{600,150}^{D} = \frac{P_{600,150}^{D} L}{bd^2}$$
(6-2)

 T_{600} = area up a deflection of L/600 (6-3)

 T_{150} = area up a deflection of L/150 (6-4)



Figure 6.9: ASTM C1609 Standard Measures of FRC toughness characterization.

However, there are number of difficulties associated with these methods, including problems around identifying the first-crack deflection, as it is frequently difficult to determine the first cracking point correctly, which leads to calculation of incorrect toughness values. Moreover, different fibre type and volume fractions show different behaviour in load-deflection curves and $10.5 \delta_1$ in the case of ASTM C1018-97 (1997) is not enough to represent the energy absorption capacity of the deflection hardening of FRGC at peak load (Haider 2014). The strain-hardening performance of fibre reinforced concrete could have a much larger deflection with a peak load higher than the first crack strength. Also, the JSCE-SF4, (1984) method is not an effective method to quantify the strain hardening performance as only the energy absorption up to 3 mm

(if the span equals 450 mm) is used, which means that the computed toughness may not then truly represent the energy absorption capacity of the material (Jiang, 2003). Moreover, this technique may be criticized for not distinguishing between the prepeak and the post-peak behaviours, and adopting an averaged approach of using the combined area under the curve to calculate the flexural toughness factors. A similar complication occurs in the ASTM C1609 / C1609M-05, (2005) Standard, as the standard code recommends use of the estimated toughness up to a net deflection of 1/150 of the span. Kim *et al.*, (2011) suggested additional points *L/100* and *L/50* to the recommended two points including L/600 and L/150 in the current ASTM C1609 / C1609M-05, (2005) to analyse the flexural behaviour of FRGC with strain hardening behaviour. ASTM C1018-97 (1997) characterized the strength remaining after cracking as residual strength factor R derived from the toughness indexes which represents the post cracking strength to the first crack strength at a given deflection interval. The residual strength factor $R_{5,10}$ and $R_{10,20}$ was recommended by ASTM C1018-97 (1997), and these parameters can be determined as below;

$$T_{3\delta}$$
 = area up to a deflection of $3\delta_1$ (6-5)

$$T_{5.5\delta}$$
 = area up to a deflection of $5.5\delta_1$ (6-6)

 $T_{10.5\delta}$ = area up to a deflection of $10.5\delta_1$

$$R_{5,10} = 100\% * \frac{(I_{10} - I_5)}{(10 - 5)}$$
(6-8)

 $\alpha \alpha$

(6-7)

$$R_{10,20} = 100\% * \frac{(I_{20} - I_{10})}{(20 - 10)}$$
(6-9)

• Ductility Indexes — A Toughness Measurement of Strain Hardening Response.

The toughness value cannot distinguish the difference between strain hardening material and softening behaviour and the level of hardening. A strain-softened FRC with high flexural strength could show a similar toughness value to strain hardening FRC with a low flexural stress. The Ductility Index (DI) is more straightforward method to determine and compare the strain hardening behaviour of fibre reinforced concrete (FRC), which is defined as below;

$$DI = \frac{\text{Deflection at Ultimate Load}}{\text{Deflection at the first cracking load}}$$
(6-10)

If DI> 1, the material shows perfect strain-hardening behaviour. The larger the value DI, the higher the ductility, and the more likely a strain-hardening response.

The flexural performance of geopolymer materials in this chapter is examined by combining the ASTM C1018-97 (1997) and ASTM C1609 / C1609M-05, (2005) standard codes with (Kim *et al.*, 2008; Kim *et al.*, 2011) recommendations in order to evaluate the effect of fibre type and volume fraction on the strain hardening performance of FRGC (Figure 6.6 and Figure 6.8). For all the examined mixtures, load, strength, energy absorption (Residual strength and toughness indexes) were determined using test results for the six points ($3\delta_1$, $5.5\delta_1$, $10.5\delta_1$, L/600, L/150, and L/100) beside the first and second peak loads. Toughness up to the peak load (T_p) and toughness peak indices (I_{peak}) (the ratio of T_p to the first cracking load-deflection area) (Shaikh, 2013a) have also been examined. Additional deflection point (δ_p) at the peak load regardless of the first-peak or second-peak was evaluated, as per ASTM C1609 / 1609M-05, (2005) (See Figure 6.10) (Kim *et al.*, 2008; Kim *et al.*, 2011; Shaikh, 2013a).



Figure 6.10: Load–deflection curve for fibre reinforced concrete and flexural performance evaluation based on ASTM C1609-05 and Kim et al.'s recommendations.

6.3.2.2 Flexural load versus deflection response of FRGC.

The primary advantage of using fibres is that they improve ductility in the load carrying capacity after cracking, which can be determined from load–deflection measurement. The effect of different volume fractions and fibre types at the same volume fraction on the load-deflection relationships are examined. The flexural responses of all examined mixtures for loads, deflections and bending strength using load-deflection relationships are summarized in Table 6.2.

6.3.2.2.1 Flexural load-deflection behaviour of geopolymer mortar without fibre.

In order to provide a basis of comparison and for control, the flexural strength of plain geopolymer mortar was investigated. Also, OPC mortar specimens were examined to compare these with the flexural performance of plain geopolymer mortar cured under ambient temperature. Three samples, without fibre, using beam specimens with the same dimensions as the fibre reinforced specimens and same mortar composition, were used. The flexural load-deflection curves recorded are plotted in Figure 6.11, and photos of specimens after testing are shown in Figure 6.12.



Figure 6.11: Flexural load deflection of plain geopolymer mortar and plain OPC mortar.

The flexural load results indicated that plain geopolymer mortar showed similar brittle behaviour to OPC mortar without fibre. The flexural load deflection linearly increased up to the ultimate load then dropped suddenly. The flexural load of plain OPC mortar and geopolymer mortar was almost the same, at 8.4 MPa. However, plain geopolymer mortar showed higher deflection than plain OPC mortar from 0.1 mm to 0.14 respectively. All the geopolymer composite without fibres failed in a brittle manner, as cracking occurred at the mid-span and propagated quickly to the top, and the specimens fractured into two pieces (Figure 6.12).



Figure 6.12: beam specimens of plain geopolymer mortar.

6.3.2.2.2 Influence of micro steel fibre on the flexural load carrying capacity of SFRGC.

The flexural load deflection relationship with varying aspect ratios (37.5 and 81.25) and volume fractions of steel fibre has been examined. The flexural load deflections of SFRGC mixtures including two aspect ratios of straight steel fibre are presented in Figure 6.13 and Figure 6.14.



Figure 6.13: Flexural load deflection of SFRGC reinforced by 2% and 3% Vf of ST6.


Figure 6.14: Flexural load deflection of SFRGC reinforced by 1%, 2% and 3% V_f of ST13.

Figure 6.13 and 6.14 compares the flexural load delection curve for samples containing 6 mm and 13 mm steel fibre length. Results using both steel fibre aspect ratios indicated that the addition of steel fibres positively affected the post cracking behaviour of all the geopolymer mixtures. The load-deflection curves of all the fibre reinforced geopolymer mixtures were similar as the load linearly increased up to the first peak, and then the nonlinear part increased up to the ultimate load. After the ultimate load points, the load deflection curves gradually decrease up to failure.



Figure 6.15: Comparison between varying aspect ratios and volume fractions of steel fibre with the strength and related deflection at first crack point (a), and maximum stress (b).

From load deflection relationships listed in Figure 6.12 and 6.13, Figure 6.15 gives a comparison of first crack and maximum stress and related deflection with varying steel fibre volume fraction and aspect ratio. A strong relationship can be detected between the steel fibre volume fraction and aspect ratio, and the first cracking and related deflection. An increase in the aspect ratio from 37.5 to 81.25 leads to an increase in the first cracking stress from 4.32 MPa to 5.4 MPa at 2% Vf, respectively while the first cracking strain increased from 0.15 mm to 0.3 mm (Figure 6.15a). The same strong trend is observed for the post cracking maximum stress as the correlations of the equations are above 92% for the proposed relationship between the maximum flexural strength and related deflection with the steel fibre volume fraction and aspect ratio (Figure 6.15b). The maximum flexural strength and related deflection of 2% V_f steel fibre increased from 4.75 MPa, 0.22 mm to 10 MPa, 1.75 mm, by increasing the aspect ratio from 37.5 to 81.25, respectively. The load carrying capacity and related deflection of SFRGC were improved dramatically by increasing the steel fibre volume fraction. The deflections at first cracking were 0.15 mm, 0.25 mm for 2ST6 and 3ST6 mixtures, respectivly. The ultimate flexural load of SFRGC was improved by 30%, and 56% for the mixture reinforced by ST6 and 75%, 175%, and 240% for the mixture reinforced by ST13 at 1%, 2% and 3% volume fraction, respectively, compared with the plain geopolymer mortar. The 3ST13 mixture showed the highest flexural strength with higher deflection capacity to about 12 MPa and 2.6 mm. The failure behaviour changed from brittle to ductile behaviour as the cracking occurred on one side and continuously and slowly increased across the sample. Photographs of the SFRGC after failure are presented in Figure 6.16.



Figure 6.16: Beam specimens of SFRGC reinforced by 2ST6 (a) and 2ST13 (b).

6.3.2.2.3 Influence of macro fibre and hybrid micro and macro steel fibre on the flexural load capacity of FRGC.

The effects of macro hooked end steel fibre incorporation, and use of a hybrid (i.e. macro hooked end steel fibre with micro steel fibre) steel fibre addition on the flexural load deflection relationship are illustrated in Figure 6.17. Steel fibre lengths of 13 mm and 0.16 mm diameter were used in combination with the macro hooked end steel fibres. Figure 6.18 shows the flexural response as a function of micro fibre content on the first crack and maximum flexural strength value and related deflections.



Figure 6.17: Flexural load deflection relationship of SFRGC reinforced by macro 1% HE steel fibre and hybrid macro with micro straight steel fibre 13mm.



Figure 6.18: Influence of macro fibres on the strength and related deflection at first crack point (a), and maximum stress (b).

The load deflection relationship clearly divided into three sections (Figure 6.17); a linear section, a strain hardening section and a strain softening section with low flexural strength to around 3.8 MPa when only macro HE fibres were utilized. Applying micro straight steel fibres with macro hooked end steel fibre in a hybrid system considerably improved the flexural load and deflections of SFRGC depending on the volume fraction of ST13. The first cracking load of 1HE-1ST13 and 1HE-2ST13 mixtures increased by 73% and 170% and the corresponding deflection increased by 100% and 250%, respectively compared with first cracking load and related deflection of the 1HE mixture. The ultimate flexural load of 1HE, 1HE-1ST13, and 1HE-2ST13 was 7.8 MPa, 14 MPa, and 25 MPa, respectively. This behaviour can be explained as the stress in the hybrid SFRGC is well distributed and its mechanical properties improved due to the short steel fibres efficiently resisting micro cracks, at the point when the micro cracks are just initiated in the geopolymer specimen. As the micro cracks develop and merge into larger macro cracks, long hooked end steel fibres become more active in crack bridging (Figure 6.19).



Figure 6.19: Beam specimens of SFRGC reinforced by 1HE (a), 1HE-1ST13 (b) and 1HE-2ST13 (c).

6.3.2.2.4 Influence of polyvinyl alcoholic (PVA) fibres on the flexural load carrying capacity of FRGC.

The flexural load deflection relationships of geopolymer material reinforced by two volume fractions (1% and 2%) of a single aspect ratio (800) PVA fibre are given in Figure 6.20 and discussed below. Each load–deflection curve in the figure is averaged from two or three specimens as previously discussed.



Figure 6.20: Flexural load deflection relationship of PVA FRGC at 1% and 2% V_f.

In the case of PVA-FRGC, the geopolymer composite showed high deflection capacity performance (Figure 6.20) and first and second peak loads can be clearly observed in the load-deflection curve. Increasing the PVA fibres volume fraction did not affect the ultimate flexural load. However, deflection at the first and second peaks significantly increased from 0.17 mm and 2.19 mm to 0.18 and 5.5 mm for 1PVA and 2PVA mixtures, respectively. The load deflection curve showed that after the initial cracking, load increased with deflection with a low slope due to the fibre bridging action at the interface of cracks, up to the point at which slip between the fibres and matrix happened. Tension softening and load moderately decreased after the second peak load at the crack surface was achieved. This is due to strong bonds between fibres and matrix. The maximum flexural load value of PVA-containing samples was close to the flexural load of the 13mm steel fibre mixture (at around 20 MPa) at the same volume fraction. In addition, the experimental test showed bendable behaviour of the composite, and the beam returned to the initial beam shape after finalization of the flexural test (Figure 6.21).



Figure 6.21: Beam specimens of 2% V_f of PVA reinforced FRGC, shown after flexural testing.

6.3.2.2.5 Influence of glass fibre on the flexural load capacity of FRGC.

The load-deflection relationship of the glass fibre reinforced geopolymer specimen is plotted in Figure 6.22. After testing of different volume fractions (0.5%, 1%, and 1.5%), it was found the 1% of glass fibre was the optimum fraction percentage in terms of sample workability, and so this fraction was used for subsequent flexural testing.



Figure 6.22: Flexural load deflection relationship of Glass-FRGC at 1% Vf.

It can be seen that glass fibre improves the peak load by 50% compared with the plain geopolymer mortar. However, the 1Glass mixture peak load is lower than steel and PVA at the same volume fraction and strain-softening failure can be recognized in the

post crack behaviour with small deflection capacity (Figure 6.11a). This is due to the weak bond between the glass fibre and the geopolymer matrix.

6.3.2.2.6 Comparisons of flexural load-deflection relationships for variant fibre types (PVA, steel, glass) at the same dosage.

In order to compare the flexural behaviour of FRGC according to the type of fibre, the load-deflection curves of steel, PVA, glass and hooked end steel fibre at 1%, 2% and 3% volume fraction are given in Figure 6.23. The first crack stress and maximum stress were determined from the load deflection curves of the test series as previously explained.





Figure 6.23: Comparisons of load-deflection relationships for variant fibre types with the same volume fraction: 1Glass, 1ST13, and 1PVA mixtures (a); 2ST6, 2ST13, 1HE-1ST13 and 2PVA mixtures (b); and 3ST13, 1HE-2ST13 and 3ST6 (c).

Figure 6.23 describes the development of flexural load resistance in the ascending range of the load–deflection curves. The load-deflection curves of all the fibre reinforced geopolymer mixtures showed a similar overall trend as the load linearly increased up to the first peak, and the nonlinear part of the curve increased up to the ultimate load. After the ultimate load points, the load deflection curves gradually decrease up to failure.

At the same volume fraction, and within this broad overall trend, the load deflection curve however deviated with the type of fibre utilized in the mixture. Figure 6.23a indicated that all of the FRGC mixtures (1PVA, 1HE, 1ST13 and 1Glass) showed strain hardening behaviour. However, the deflection hardening was less pronounced with glass fibre reinforced geopolymer concrete. The 1PVA mixture showed the highest first crack and ultimate stress load followed by 1ST13, 1Glass and 1HE mixtures, respectively (Figure (6.24 a-b)). The deflection capacities at the first crack stress were 0.17 mm, 0.14 mm, 0.24 mm, and 0.75 mm for 1HE, 1PVA, 1Glass and 1ST13 mixtures, respectively. The deflection of the 2PVA mixture at the second peak load is much higher than all geopolymer composites containing glass, micro and macro steel fibre (Figure 6.24b). The load carrying capacity and related deflection of the fibre reinforced geopolymer concrete were improved dramatically by increasing steel fibre

dosage rates and aspect ratios (Figure 6.24 (b and d)). The 2ST13 mixture showed the highest load carrying capacity at the first crack and maximum post cracking, at 5.4 MPa and 10 MPa, respectively. The maximum flexural load value of PVA-containing samples was close to the flexural load of the 13mm steel fibre mixture (at around 20 MPa) at 2% volume fraction. Increasing straight steel fibre length from 6 mm to 13 mm increased the ultimate flexural load by 100% at the same volume fraction. The 2ST13 mixture showed the highest deflection at first crack strength. However, the 2PVA mixture showed a superior deflection capacity at the maximum strength followed by 2ST13, and 2ST6 mixtures, respectively. The same trend was observed at 3% volume fraction, where the peak load deflections of the 3ST13 mixture provide almost 3 times higher deflection than the 3ST6 mixture.



Figure 6.24: Influence of discontinuous fibre type on load carrying capacity of FRGC.

As can be seen from Figure 6.21 and Figure 6.23, all fibre reinforced geopolymer mixtures apart from the 2ST6 mix confirm strain hardening behaviour as the second peak load is greater than that at the first cracking load. The level of hardening is determined by examining the flexural toughness, toughness indices and ductility factors in the next section.

6.3.2.3 Flexural toughness response of FRGC.

Both ASTM C1018-79 and ASTM C1609/C1609-05 standards are reliable and suitable for evaluation of the ductility of fibre reinforced geopolymer concrete, as discussed previously. Therefore, flexural toughness tests of fibre reinforced geopolymer concrete following these standards have been carried out in this study. For all the examined mixtures, energy absorption (Residual strength and toughness) quantities of the test results for the six points $(3\delta_1, 5.5\delta_1, 10.5\delta_1, L/600, L/150, and L/100)$ beside the first and second peak loads are presented. Toughness up to the peak load (T_p) and toughness peak indices (I_{peak}) (the ratio of T_p to the first cracking load-deflection area) (Shaikh, 2013a) have also been examined. Additionally, ductility indexes (ratio of deflection at the peak load to deflection at first-peak point) are examined to evaluate strain hardening behaviour.

		2ST6	3ST6	1 ST13	2ST13	3ST13	3[ST6-ST13]	1HE	1HE-1ST13	1HE-2ST13	1 PVA	2PVA	1Glass
	P (KN)	9.6	10.0	9.6	12.0	14.5	14.0	5.5	9.5	14.8	10	11	10
first crack	δ (mm)	0.2	0.3	0.2	0.3	0.6	0.4	0.2	0.3	0.6	0.1	0.2	0.2
(δ)	F (MPa)	4.3	4.5	4.3	5.4	6.5	6.3	2.5	4.3	6.7	4.5	5.0	4.5
	T (joule)	0.6	1.3	1.0	1.8	4.3	2.8	0.5	1.6	4.4	0.7	1.0	1.2
	P (KN)	10.2	12.7	14.0	18.0	25.0	25.5	7.1	13.5	24.4	13	15	13
25	δ (mm)	0.4	0.8	0.6	0.9	1.8	1.2	0.5	1.0	1.8	0.4	0.5	0.7
30	F (MPa)	4.6	5.7	6.3	8.1	11.3	11.5	3.2	6.1	11.0	5.9	6.8	5.8
	T (joule)	3.0	7.0	5.7	10.7	28.0	18.6	2.6	9.4	28.0	4.0	5.7	6.7
	P (KN)	10.0	10.5	13.4	22.0	25	22.0	7.2	14.0	20.0	15	17	11
5.55	δ (mm)	0.7	1.4	1.1	1.7	3.3	2.2	0.9	1.9	3.3	0.8	1.0	1.3
5.50	F (MPa)	4.5	4.7	6.0	9.9	11.3	9.9	3.2	6.3	9.0	6.8	7.4	5.0
	T (joule)	6.0	14.2	12.5	25.7	65.0	43.0	5.6	21.1	61.3	8.8	16	13.8
	P (KN)	7.0	6.0	11.8	18.6	11.5	8.0	6.2	11.5	15.2	18	18	4.3
10.58	δ (mm)	1.3	1.6	2.1	3.2	6.3	4.2	1.8	3.6	6.3	1.5	1.9	2.5
10.50	F (MPa)	3.2	2.7	5.3	8.4	5.2	3.6	2.8	5.2	6.8	8.0	8.1	1.9
	T (joule)	11.0	16.3	25.7	56.2	121.0	74.4	11	42.7	115.2	19	28	23
	P_{600}^{100} (KN)	9.5	12.7	14.0	16.5	16.0	19.4	7.5	12.5	17.0	15	16	13
L/600= 0.75mm	f (MPa)	4.3	5.7	6.3	7.4	7.2	8.7	3.4	5.6	7.7	6.7	7.2	5.8
	T_{600} (joule)	6.6	7.0	7.8	8.1	6.2	7.1	4.0	4.7	6.5	8.3	9.0	7.0
	Pp (KN)	10.5	12.8	14.0	22.0	26.9	16.0	7.8	14.0	25.0	19	20	13
Second neak	δ (mm)	0.2	0.6	0.8	1.8	2.6	1.5	0.8	1.8	1.8	2.2	5.5	0.8
Second peak	F (MPa)	4.7	5.8	6.3	9.9	12.1	7.2	3.5	6.3	11.3	8.5	8.9	5.9
	Ţ (joule)	3.2	5.3	7.8	28.6	48.0	58.0	4.3	19.7	27.0	31	84	6.9
	P_{150}^{100} (N)	3.0	5.0	9.7	18.8	26.0	22.0	6.6	12.5	20.0	12	18	3.0
L/150=3mm	f (MPa)	1.4	2.3	4.4	8.5	11.7	9.9	3.0	5.6	9.0	5.2	8.1	1.4
	T (joule)	18.9	26.5	35.3	56.0	60.0	43.0	25	36.0	53.9	19	48	23
	P_{100}^{100}			7.0	13.2	19.5	6.5	4.6	10.0	16.2		19	
L/100=4.5mm	f (MPa)			3.2	5.9	8.8	2.9	2.1	4.5	7.3		8.6	
	T_{600} (Joule)			47.9	80.0	94.1	64.4	33	52.9	81.0		76	
	I5	3.9	5.6	5.9	6.1	6.5	6.6	5.5	5.8	6.3	5.6	5.7	5.6
Toughness	I10	8.1	11.4	11.8	14.6	15.0	15.4	12	13.1	13.7	17	13	10
Indices ASTM	I20	15.2	19.7	26.7	31.9	27.9	26.6	24	26.4	25.9	26	29	17
C1180	R _{5.10}	84.2	115.2	117	170.5	170.5	174.3	129	144.2	148.6	224	144	89.3
	R _{10,20}	71	83.2	149	173.3	129.0	112.1	121	133.7	122.1	95.6	156	73.9
indexes peak	I peak	5.3	4.2	8.1	16.2	11.1	20.7	9.1	12.2	6.1	42.9	84.8	5.8

Table 6.2: Flexural response of fibre reinforced geopolymer composite.

6.3.2.3.1 Flexural toughness following ASTM C1018 standards.

The toughness (representing the area under the load deflection curve up to a given deflection point at $3\delta_1$, $5.5\delta_1$ and $10.5\delta_1$) and toughness indices I₅, I₁₀ and I₂₀ were used as mentioned previously to identify the toughness capacity of FRGC with different fibres (Table 6.2 and Figure 6.25).



Figure 6.25: Toughness up to $\delta 1$, $3\delta 1$, $5.5\delta 1$, $10.5\delta 1$ deflection points of FRGC, with varying aspect ratio and volume fraction of steel fibre (a); macro and combined macro with micro steel fibre (b); and PVA and glass fibre (c).

Table 6.2 and Figure 6.25 present the toughness and toughness indexes at (δ_1 , $3\delta_1$, $5.5\delta_1$, $10.5\delta_1$) deflection points according to ASTM C1018-97, (1997). The toughness value increased at higher deflection value for all the examined mixtures. As shown in Figure 6.25a, the toughness at all deflection points increased with increasing aspect ratio and volume fraction of steel fibre. The 3ST13 mixture showed the highest toughness value at around 4.4 joule, 28 joule, 66 joule, and 120 joule at δ_1 , $3\delta_1$, $5.5\delta_1$, $10.5\delta_1$, respectively. Inclusion of macro hooked end steel fibres showed low toughness values of around 2.6 joule, 5.6 joule, and 11.3 joule at $3\delta_1$, $5.5\delta_1$, $10.5\delta_1$, respectively. However, use of a hybrid macro HE and micro steel fibre mix considerably improved

the toughness, as toughness at 10.5 δ_1 deflection point of 1HE-1ST13, 1HE-2ST13 mixtures was 3 times and 9 times higher than corresponding toughness value of 1HE mixture, respectively (Figure 6.25b). The toughness of PVA mixtures showed similar toughness values at 1% volume fraction. However, toughness of the 2PVA mixture was much lower than the 2ST13 mixture up to $10.5\delta_1$ deflection point. This is due to small deflection value at first crack, and $10.5\delta_1$ equal to 1.9 mm which it is too far from the deflection capacity at the peak load around 5.5 mm. Thus, toughness up to 10.5 times of the first crack deflection is not enough to represent the total toughness of the strain hardening capacity of fibre reinforced geopolymer concrete. The toughness at higher deflection was examined by following ASTM C1609 / C1609M-05, (2005) as discussed earlier.



Figure 6.26: Toughness indexes I5, I10 and I20.

The toughness indexes represent the stiffness after first crack up to the particular end point deflection. As shown in Figure 6.26, the toughness indices are almost the same in all the examined fibres at the small deflection value up to 3 times the first-crack deflection. However, the toughness indices of the geopolymer concrete clearly increased when steel fibre reinforcements were used. The flexural toughness of the SFRGC increased with increasing aspect ratio of the steel additive: Toughness indices I_{20} increased from 19 for the ST6 mixture to 32 for ST13. However, increasing the short straight steel fibre dosage rate did not generate significant differences in flexural toughness indexes values. The results also indicated that the toughness indexes I_5 , I_{10} and I_{20} were close to values of 5, 19 and 20 respectively for all examined mixtures which mean the material behaviour can be approximated to perfectly plastic after the first crack. All FRGC mixtures exhibited very high I_{20} values in excess of 20, apart from the 2ST6 mixture, which is even superior to perfectly plastic behaviour. According to Naaman, (2003a) the I20 > 20 is an indication of deflection hardening behaviour in fibre composites. In addition, the residual strength factors, which can be determined from the toughness indexes, are presented in Table 6.2. The results showed that the $R_{5,10}$ and $R_{10,20}$ values of all FRGC mixture apart from ST6 and Glass fibre reinforced mixtures were higher than 100 corresponding to perfectly plastic behaviour. Thus, it can be concluded that all ST13, HE and PVA fibre reinforced mixtures exhibited considerably improved post-cracking flexural performance, which is even superior to perfectly plastic behaviour.

6.3.2.3.2 Flexural toughness following ASTM C1609 standard codes.

Toughness and residual strength have been determined at L/600 and L/150 deflection points according to ASTM standard C1609, with the addition of point L/100 to cover the total area of the load deflection curve (Table. 6.2 and Figure 6.27).



Figure 6.27: Toughness and residual strength at deflections L/600 and L/150, with varying aspect ratio and volume fraction of steel fibre (a); macro and combined macro with micro steel fibre (b); and PVA and glass fibre (c).

As shown in Figure 6.27, the toughness values of different fibre reinforced specimens at low deflection value (L/600 = 0.75mm) are almost the same for all the examined mixtures. The lowest values of toughness T_{600} (4.7 joule) were shown for the 1HE-1ST13 mixture. The residual flexural strength f_{600} , f_{150} and the toughness parameters T_{150} were considerably altered by utilizing different types of fibre. The toughness of SFRGC before and after the second peak load was also considerably influenced by fibre volume fraction and aspect ratio. The experimental results indicated that the aspect ratio of the steel fibres influenced the post-crack behaviour, as with increasing steel fibre volume content and aspect ratio, an increase in the toughness values is observed. The residual strength f_{150} increased from 35 MPa for the 1ST13 mixture to 56 MPa and 60 MPa for 2ST13 and 3ST13 mixtures, respectively (Figure 6.27a). Fibre reinforced geopolymer 3ST13 gave the highest toughness T₁₅₀ value of 60 joules. When combining two steel fibre lengths (of 13mm and 6 mm), the toughness values were close to the toughness of the 3ST13 mixture. The toughness of hooked end fibre reinforced geopolymer mixtures increased with the inclusion of straight 13mm steel fibre. Toughness T_{150} of the HE fibre mixture increased with increasing straight steel fibre content from 36 joules to 54 joules for 1HE-1ST13 to 1HE-2ST13, respectively (Figure 6.27b). Inclusion of PVA fibre in the geopolymer mixture considerably enhanced the post cracking toughness and increasing PVA fibre volume fraction from 1% to 2% V_f increased the post cracking toughness values T₁₅₀ from 19 joules to 48 joule. However, increasing the PVA fibre volume fraction did not considerable change the residual strength. This indicates that increasing the PVA fibre volume fraction enhanced the strain hardening behaviour, which can be evaluated by toughness and toughness indexes at the peak strength (Figure 6.28) and ductility indexes.

6.3.2.3.3 Flexural toughness and toughness indexes up to peak load.

Toughness and toughness indices up to the peak load were also examined to evaluate the effect of fibre types and aspect ratios on the ultimate load-deflection values (Figure 6.28).



Figure 6.28: Average flexural toughness of SHGC: Peak load toughness (Tp) and peak toughness indices Ipeak (b).

For non-reinforced geopolymer concrete, toughness indices are taken as 1.0 due to the plain mortar flexural test specimens failing directly after the formation of the first crack. Toughness indices Ipeak and peak toughness increased with increasing steel fibre volume fractions and aspect ratios. This is due to higher frictional surface area between the steel fibres and the geopolymer matrix, resulting in stronger crack bridging by the long steel fibres and thus a more efficient load transfer mechanism. Toughness, and toughness indices, at the peak load of PVA fibre reinforced geopolymer significantly improved by increasing the fibre volume fraction from 1% to 2%. 2PVA-FRGC exhibited highest toughness indices values of 84 at I_{peak} proving the superior deflection hardening behaviour of this composite material. The higher the Ipeak value the more non-elastic energy absorption capacity of the composite. The strong bond between PVA and steel fibre with the geopolymer matrix is the major toughening mechanism in the geopolymer composite as indicated by the microstructural analysis (SEM section, below) in this study. Toutanji et al., (2010) have noted that adding fibres to the matrix changes the behaviour from plain linear, where the energy absorption capacity relates to fracture and initiation of cracking, to a ductile behaviour, where the energy absorption is measured by micro-crack development and progressive fibre debonding up to failure.

6.3.2.3.4 Ductility Indexes (DI).

To provide a general idea of the comparative strain hardening performance of different fibre reinforcement geopolymer mixtures, the ratio of deflection at the peak load to that at first crack (ductility index, DI) for all series has been illustrated in Figure 6.29. The deflection hardening behaviour of fibre reinforced cementitious mixtures is confirmed if the peak load and related deflection is greater than that the comparative values at the first cracking point.



Figure 6.29: Ratios of peak load to first crack load and deflection at peak load to that at first crack (ductility index, DI) of all FRGC.

Most of the FRGC mixtures exhibited a P_p/P_1 ratio of more than 1 (See Figure 6.29). The experimental results showed that the ductility indexes of SFRGC mixtures increased by increasing the fibre length and aspect ratio of straight steel fibre. The ductility indexes of 2ST13 and 3ST13 mixtures increased by 280% and 77% compared to the short steel fibre (6mm length) at the same volume fraction. The large gap between the ultimate load and the load at the first crack and higher ductility indexes of FRGC mixtures ensures the deflection hardening performance. The greatest gap between deflections at the peak load to that at first crack load which indicated the highest deflection hardening capacity was obtained in fibre reinforced geopolymer reinforced by using 2% volume fraction of PVA, followed by 1PVA and 2ST13.

6.3.3 Direct tensile strength test.

Direct tensile tests were carried out in order to examine the effect of fibre types, aspect ratios, volume fractions and curing time on the tensile strength of the examined mixes. Direct tensile strength was determined using 'dog bone' shaped samples of 13mm (mid cross section) by 50 mm. The testing set up used has been described previously in Chapters 3 and 5.

6.3.3.1.1 Tensile strength development of FRGC over the curing time.

The effect of curing age on the tensile stress-strain relationship of FRGC was evaluated by testing geopolymer specimens at 3, 7, 28 and 90 days curing age (Figure 6.30 and Table 6.3).











Figure 6.30: Effect of curing time on the stress strain relationship under tensile strength: 2ST mixtures (a); 2ST13 mixtures (b); 3ST13 mixture (c); 3 hybrid 3(ST6-ST13) mixture (d); 2PVA mixture (e); 1Glass mixture (f).

		Tensile Strength [MPa]				
Mix ID	3 days	7 days	28 days	90 days		
	0.70	0.86	1.60	2.33		
2ST6	0.70	0.76	1.73	2.65		
	0.69	0.90	1.80	2.38		
	NA	0.88	2.55	NA		
3ST6	NA	0.79	2.33	NA		
	NA	1.00	2.40	NA		
	1.60	NA	2.40	NA		
1ST13	1.40	NA	1.80	NA		
	1.80	NA	2.20	NA		
	0.79	1.45	2.46	3.46		
2ST13	1.01	1.22	3.01	3.27		
	0.80	1.20	2.57	3.40		
	1.73	2.50	3.55	3.67		
3ST13	1.68	2.59	3.31	3.97		
1	1.66	2.55	3.40	3.54		
	NA	2.03	2.23	2.34		
3[ST6-ST13]	NA	2.46	2.46	2.79		
	NA	2.06	2.20	2.51		
	1.60	2.00	2.50	NA		
1PVA	1.40	1.80	2.60	NA		
	1.80	2.30	3.00	NA		
	NA	2.26	3.24	NA		
2PVA	NA	2.14	3.43	NA		
	NA	2.00	3.80	NA		
	NA	1.28	3.13	NA		
1Glass	NA	1.68	3.26	NA		
	NA	1.80	2.04	NA		

Table 6.3: Ultimate tensile strength versus curing time.

Figure 6.30 and Table 6.3 showed that curing time significantly affected the ultimate tensile strength and post cracking behaviour in the stress-strain curves. At early ages (3 and 7 days), tensile performance of the 2ST6 mixture cured under ambient temperature is about 0.7 MPa and 0.85 MPa, respectively (Figure 6.30a). This early strength is low compared to the specimens reinforced with 2ST13 (at about 1.3 and 1.43 MPa, respectively) (Figure 6.30b). 2PVA-FRGC specimens show higher tensile strength than all specimens reinforced with both aspect ratios of steel fibre. At 7 days, the tensile strength of 2PVA-FRGC is about 2.13 MPa, which is greater than the tensile strength of SFRGC (which is 0.85 MPa and 1.3 MPa for 6 mm and 13 mm fibre length, respectively) (Figure 6.30e). This is due to a relatively strong early interfacial bond forming between the PVA fibres and the geopolymer matrix compared with the steel fibre reinforced composite. For all SFRGC specimens, the tensile strength was significantly improved after 7 days. The results show the same trend with high values for all geopolymers reinforced with different fibres observed compared to their respective values at early ages (Table 6.3). At 28 days tensile strength increased by 104%, 95%, 65% and 77% for 2ST6, 2ST13, 2PVA and 1Glass mixtures, respectively compared to the respective mixtures at 7 days. Under room temperature curing, geopolymerization improved with curing time, leading to improvements in the strength of the geopolymer matrix, and enhancement of the bond between the geopolymer matrix and the reinforcement fibres. Moreover, curing time also influenced the stress-strain curve and the post cracking strength and energy absorption. The areas under the stress-strain curves of FRGC up to 2% strain are presented in Table 6.4. For all FRGC mixtures, increasing the curing time lead to an increase in the energy absorption. However, the type of failure was still the same for all curing ages, which is a softening strain failure for short length of steel fibre and strain hardening failure for PVA and ST13 fibre reinforced geopolymer mixtures.

Table 6.4:	Tensile energy	absorption	with curing	time.

Age	2ST6	3ST6	3[ST6-ST13]	2ST13	3ST13	1GLASS	1PVA	2PVA
3 days	0.008			0.012	0.024		0.007	
7 days	0.010	0.004	0.023	0.014	0.034	0.021	0.015	0.029
28 days	0.017	0.012	0.031	0.040	0.048	0.018	0.033	0.048

6.3.3.1.2 Effect of fibre type, aspect ratio and volume fraction on the tensile strength performance of FRGC.

The tensile stress-strain curves for FRGC reinforced with varying fibre types, aspect ratio and volume fractions are given in Figure 6.31 and Figure 6.32. Figure 6.31 illustrates the tensile stress strain behaviour of plain geopolymer mortar (a), and FRGC containing 2% and 3% volume fraction of short straight steel fibre (6mm) (b); 1%, 2% and 3% volume fraction of long straight steel fibre (13mm) (c); 1% and 2% volume fraction of PVA (d) and 1% glass fibre reinforced geopolymer (e). The effect of fibre types on the tensile performance at the same volume fraction is illustrated in Figure 6.32. Figure 6.32 shows a comparison of the tensile stress-strain relationships of FRGC reinforced by 1% Glass, 1% ST13, and 1% PVA mixtures (a); 2% ST6, 2% ST13 and 2% PVA mixtures (b); and 3% ST13, 3% ST6 and 3% hybrid ST6-ST13 mixtures (c).





Figure 6.31: Tensile stress-strain relationships of FRGC with varying fibre volume fractions; plain geopolymer (a), ST6 mixtures (b), ST13 mixtures (c), PVA mixtures (d) and 1 Glass mixture (e).



Figure 6.32: Stress-strain relationships of FRGC for varying fibre types at the same volume fraction; 1% Vf mixtures (a), 2% Vf mixtures (b) and 3% Vf mixtures (c).

In order to evaluate the effect of inclusion of discontinuous fibres and to provide a basis of comparison, the direct tensile strength of plain geopolymer mortar was also investigated. The optimum ternary mixture containing (40% GGBS, 10USF and 50% FA, Chapter 5), without fibre, was tested using small dogbone samples described in the previous Chapters 2 and 3.

The tensile stress-strain relationship of the plain geopolymer mortar (See Figure 6.31a) is represented by linear build up to the ultimate stress, which then drops suddenly after formation of the first crack. However, the initial linear elastic phase of steel fibre reinforced geopolymer concrete is followed by a non-linear strain-hardening behaviour up to the peak load only in the case of specimens with 13mm long steel fibres or hybrid specimens with 6mm and 13mm long fibres. In specimens with 6mm length steel fibre only, there was no strain hardening after the linear part of the curve, which is attributed to the lower surface friction between the geopolymer matrix and the short steel fibres (Figure 6.31). It is clearly observed that increasing the steel fibre volume fraction considerably affects the post cracking stress and strain failure behaviour for both aspect ratios. The highest strength and strain hardening behaviour in the post peak stress values of SFRGC mixtures was achieved in the specimens using 13mm length fibres, where a longer friction surface between the fibres and the matrix is provided (Figure 6.32c).

Inclusion of PVA and glass fibres in the FRGC showed different performance from SFRGC (Figure 6.31 (d and e)). PVA-FRGC samples displayed strain-hardening behaviour accompanied by multiple cracking behaviour. Strain hardening was observed after first cracking and the stress-strain curve was almost horizontal. As displacement increased, PVA fibres started to be fractured; the strain-hardening trend gradually disappeared. On the other hand, the effect of glass fibre on the post crack behaviour of fibre reinforced geopolymer composites was less pronounced, and soft hardening failure occurred after the ultimate tensile strength was reached.

The failure pattern and crack distribution of FRGC vary under tension loading with the different fibres used. In the case of steel and glass fibre, most of the specimens failed by initiation of cracks in one or two locations in the narrow cross section of the specimens, and the crack opening became wider up to failure. Different behaviour was seen within PVA-FRGC samples as multiple cracks were noticed during loading, the strain increased at the same stress value and many of the microcracks closed after unloading the specimens. This multiple cracking improves the composite characteristics in terms of ductility, fracture energy, strain hardening, and deformation capacity under tension and compression (Matsumoto and Mihashi, 2003).

6.3.3.2 Tensile stress-strain models of fibre reinforced geopolymer concrete

Based on the tensile stress-strain performance of varying FRGC mixtures with strain hardening or softening behaviour (Figure 6.31 and Figure 6.32), two stages of FRGC response before destruction under tension load are defined in Figure 6.33. The first stage represents the stress distribution during the elastic range before first cracking E, and the second stage corresponds to the post-cracking modulus E_{cr} .



Figure 6.33: Effect of fibre on tensile bond characteristics of FRGC.

The effect of fibre type, volume fractions and aspect ratios on the tensile bond characteristics results of steel, PVA and glass fibres are shown in Table 6.5, and Figure 6.34. The results include the first cracking stress and maximum stress, the corresponding strain, and the elastic modulus of the FRGC.

	First crack	Energy at	Ultimate	Energy at	Е	E _{cr}
	stress	first crack	stress	ultimate stress		
	[MPa]	[MPa]	[MPa]	[MPa]	[MPa]	[MPa]
2ST6	1.4	0.000124	1.7	0.0004	9855	
3ST6	1.8	0.000081	2.3	0.0002	19978	
1ST13	1.5	0.000115	2.1	0.0015	9804	431
2ST13	1.9	0.000409	2.6	0.0030	16064	285
3ST13	2	0.001000	3.2	0.0070	24885	317
1PVA	2	0.000124	2.4	0.0020	16129	235
2PVA	2.5	0.000403	3	0.0040	23974	4414
1GLASS	1.5	0.000358	2.1	0.0021	15353	353

Table 6.5: Bond characteristics of fibre reinforced geopolymer concrete.



Figure 6.34: Comparison of different properties of FRGC: first cracking stress and energy at first cracking stress (a), maximum stress and energy at post cracking stress (b).

Figure 6.34 illustrates the variation in the tensile strength testing results of various discontinuous fibre reinforced geopolymers. Three key observations can be made from the results; (1) the first cracking stress of PVA reinforced mixtures is higher than that in steel fibre reinforced mixtures; (2) increasing the volume fraction, and aspect ratio of steel fibre leads to an increase the first cracking tensile strength, maximum stresses and related energy absorption capacity; (3) increasing the volume fraction of PVA fibre from 1% to 2% considerably improved the first cracking and maximum stress.

As shown in Table 6.5, the elastic behaviour of fibre-reinforced geopolymer is considerably affected by the fibre type and volume fraction used in the geopolymer mixture. The modulus of elasticity of 2ST13 was almost 60% higher than in the 2ST6 mixture. Increasing the volume fraction of steel fibre from 2% to 3% considerably improved the elastic modulus to 16 GPa and 23.9 GPa for the ST13 mixture, respectively. Inclusion of PVA fibres in the geopolymer mixture gave a high elastic modulus, and increasing the PVA volume fraction from 1% to 2% increase the elastic modulus from 16 GPa to 24 GPa.

After the elastic modulus, the stress-strain relationships indicate yielding of FRGC in non-linear mode, defined as a plastic state of the material. The nonlinear region is a function of the microcracks formed in the geopolymer binding matrix. The geopolymer reaches maximum stress through nonlinear strain hardening followed by strain softening once micro-cracks accumulate in a significant amount in the concrete. According to the experimental stress-strain relationships of FRGC, a low volume fraction of short steel fibre reinforced geopolymer performed as a strain softening material. However, increasing steel fibre length from 6mm to 13mm changed the geopolymer composite performance to a strain hardening behaviour. The effect of PVA fibres on load carrying capacity after cracking of FRGC is noticeable, as shown in Figure 6.31 and Figure 6.32. The post cracking modulus of 2PVA fibre reinforced geopolymer concrete is 14 times higher than in the 2ST13 mixture. Furthermore, the residual tensile strength at the plateau zone is not the same for different fibre types. This behaviour illustrates that geopolymer reinforced with PVA, 3ST13 and hybrid ST13-ST6 exhibited higher strain hardening with the geopolymer matrix than glass and short steel fibre.

6.3.3.3 Analysis – Prediction of stress-strain tensile response of FRGC.

To predict the tensile behaviour of fibre reinforced geopolymer concrete, the method proposed by Vandewalle, (2003) was used in approximating the tensile stress-strain parameters based on the stress deflection data established from flexural beam tests of the same mixtures. Figure 6.35 represents the bi-linear uniaxial stress strain diagram up to the ultimate tensile strength.



Figure 6.35: σ - ϵ Diagram for fibre reinforced concrete up to the ultimate strength.

The points defined in Figure 6.35 are measured from the following relations:

$$\sigma_1 = C_1 \cdot (1.6 - h) \cdot f_{ctm, fl} \tag{6-11}$$

$$\sigma_2 = C_1 \cdot f_{fl.ult} \tag{6-12}$$

$$\varepsilon_2 = \varepsilon_1 + 0.01\%_0 \tag{6-13}$$

Where $f_{ctm,fl}$, $f_{fl.ult}$ and E_c are the first crack and ultimate flexural tensile strength and modulus of elasticity of fibre reinforced geopolymer concrete (in MPa), respectively, h is the depth of the flexural beam (in m), ε_1 and ε_2 represent the first crack strain which is obtained from experimental work and ultimate tensile strains, respectively.

In the case of geopolymer materials, there is currently no relationship available for predicting the tensile strength of FRGC. Using the $f_{ctm,fl}$ and f_{ult} values obtained from flexural experimental tests, the tensile stress strain diagram of geopolymer concrete reinforced with different aspect ratios, volume fractions and types of straight fibres was characterized following RILEM TC 162-TDF recommendations (Vandewalle, 2003).

By comparing the experimental results with the obtained results using the constitutive law proposed by RILEM TC 162-TDF and the proposed coefficients (C_1 =0.52 and

 $C_2=0.36$), higher predicted tensile values than those recorded in the experimental results at first cracking and post cracking stress were obtained (Figure 6.36). A trial was employed to adjust stress coefficients C_1 and C_2 from the values recommended by RILEM TC 162-TDF(Barros, 2004) (Vandewalle, 2003) using the experimental data to achieve a better fitting tensile behaviour. New relations are included in Table 6.6.

The strain at the ultimate tensile value considerably changed with fibre content and aspect ratios. The ultimate strain increased with increasing aspect ratio and volume fraction of steel and PVA fibres. The largest gap between the first crack strain and the peak tensile strain was determined in the 3ST13 and 2PVA mixtures, which confirms the strain hardening performance of these mixtures. The relation was calculated and plotted between the experimental and the predicted bi-linear tensile strength values as shown in Table 6.6 and Figure 6.37.

Mix ID	First crack	Ultimate strength			
	Stress	Strain	Stress		
2ST6	$\sigma_1 = 0.24 \cdot (1.6 - h) \cdot f_{\text{ctm,fl}}$	$\epsilon_2=\epsilon_1+0.020\%$	$\sigma_2 = 0.35 \cdot f_{fl,ult}$		
3ST6	$\sigma_1 = 0.24 \cdot (1.6 - h) \cdot f_{\text{ctm,fl}}$	$\epsilon_2 = \epsilon_1 + 0.015\%$	$\sigma_2 = 0.38 \cdot f_{fl,ult}$		
1ST13	$\sigma_1 = 0.24 \cdot (1.6 - h) \cdot f_{\text{ctm,fl}}$	$\epsilon_2=\epsilon_1+0.015\%$	$\sigma_2 = 0.32 \cdot f_{fl,ult}$		
2ST13	$\sigma_1 = 0.24 \cdot (1.6 - h) \cdot f_{\text{ctm,fl}}$	$\epsilon_2=\epsilon_1+0.250\%$	$\sigma_2 = 0.26 \cdot f_{fl,ult}$		
3ST13	$\sigma_1 = 0.20 \cdot (1.6 - h) \cdot f_{\text{ctm,fl}}$	$\epsilon_2=\epsilon_1+0.550\%$	$\sigma_2 = 0.26 \cdot f_{fl,ult}$		
1PVA	$\sigma_1 = 0.20 \cdot (1.6 - h) \cdot f_{\text{ctm,fl}}$	$\epsilon_2 = \epsilon_1 + 0.200\%$	$\sigma_2 = 0.28 \cdot f_{fl,ult}$		
2PVA	$\sigma_1 = 0.34 \cdot (1.6 - h) \cdot f_{\text{ctm,fl}}$	$\varepsilon_2 = \varepsilon_1 + 0.400\%$	$\sigma_2 = 0.32 \cdot f_{fl,ult}$		
1Glass	$\sigma_1 = 0.24 \cdot (1.6 - h) \cdot f_{ctm, fl}$	$\epsilon_2=\epsilon_1+0.025\%$	$\sigma_2 = 0.36 \cdot f_{fl,ult}$		

Table 6.6: Proposed stress-strain relationships for FRGC.



Figure 6.36: Comparison of tensile stress between experimental, proposed and numerical using σ - ε method proposed by RILEM TC 162-TDF.



Figure 6.37: Experimental and modelled stress-strain responses of SFRGC.

6.3.3.4 Comparison performance of flexural strength and tensile strength tests in SHGC.

It is well known that there is a strong correlation between tensile and flexural strength for conventional fibre reinforced concrete (Xu and Shi, 2009; Kim *et al.*, 2011), and FRC with strain hardening behaviour under tension leads to deflection hardening

behaviour under flexural stress (Naaman, 2003b; Kim *et al.*, 2010; Kim *et al.*, 2011). In general, tensile strength can either be determined by splitting tensile strength, direct tensile test or flexural strength tests. Comparison between the flexural test and direct tensile strength data at the first cracking load and maximum load of fibre reinforced geopolymer concrete was carried out (Figure 6.38).



Figure 6.38: Comparison of flexural and tensile strength; strength at the first crack (a); at the ultimate strength (b).

The results show that direct tensile strength values are in broad agreement with the flexural strength performance (Figure 6.38a). However, FRGC shows a higher bending strength values which permits the load to be applied normal to the loaded surface of the beam comparable to the tensile strength values across the geopolymer material. The main conclusions that can be drawn from the experimental results is that there is an agreement between the tensile strength and flexural strength behavior for all the examined fibre reinforced geopolymer concrete mixtures. The first crack tensile strength and first crack flexural strength increased when the fibre volume fraction increased and, at the same fibre content, when the fibre aspect ratio increases. The 3ST13 mixture shows the highest first crack flexural and tensile strength followed by 2ST13 and 2PVA mixtures over all the examined mixtures. Figure 6.38b illustrates that the ultimate flexural and tensile strength is in agreement with the first crack strength results with the different fibres examined. Increasing the fibre aspect ratio and volume fraction leads to an increase in the gap between the first and second peak of the flexural and tensile strength, and improves the strain hardening behavior of the fibre reinforced geopolymer concrete. To conclude, both tensile and flexural data confirm strain hardening behavior for the 2ST13, 3ST13 and 2PVA geopolymer mixtures.

6.3.4 Microstructure Analysis of FRGC.

Scanning electron microscope (SEM) examination of fibre surface texture and fibrematrix interfaces was carried out in order to evaluate the fibre-geopolymer matrix bond characteristics. SEM images of fibre reinforced geopolymer samples were taken of cracked samples after the end of tensile testing, and are shown in Figure 6.39, Figure 6.40 and Figure 6.41. The fractured samples were sputter coated with carbon and imaged using a Zeiss, model LEO 1455VP, SEM. Figure 6.39 shows SEM images perpendicular to the fracture surface of FRGC specimens at low magnifications.



Figure 6.39: SEM images perpendicular to the fracture surface area of FRGC after tension; Glass-FRGC (a), SFRGC (b), and PVA-FRGC (c).

It can be observed from Figure 6.39, steel and PVA fibres were homogeneously dispersed in the geopolymer matrix, and a significant amount of these fibre were well attached to the geopolymer matrix. The glass fibre sample however showed heterogeneous dispersion in the geopolymer matrix and poor anchoring, which might be attributed to the smaller embedding length of the glass fibre in the geopolymer matrix. Several voids and cracks were observed in the geopolymer matrix of the fracture surface due to the pulling out of fibres and tensile failure cracking. The surface

of the fibres was evaluated by examining the specimens at higher magnification as shown in Figure 6.40 and Figure 6.41.



Figure 6.40: Sequences of focusing SEM images on the PVA fibre at the fracture surface area of PVA-FRGC.



Figure 6.41: SEM micrographs of FRGC specimens, image perpendicular to the fracture surface of steel fibre/geopolymer composites (a); image perpendicular to the fracture surface of PVA fibre/geopolymer composites (b); and image perpendicular to the fracture surface of glass fibre/geopolymer composites (c).

SEM photographs of the fracture surfaces of the FRGC after the completion of tensile tests are shown in Figure 6.41 (a-c). It can be observed that the geopolymerization process has successfully occurred and all the examined samples had a similar well-connected geopolymer matrix regardless of the fibre type used. This mixture represents an optimum ternary geopolymer mixture based on resulted obtained in Chapter Five. Combining slag as a source of calcium, with fly ash and silica fume as

source of silica leads to an increase in the formation of the geopolymerization product of a calcium alumino-silicate hydrate (C–A–S–H) gel, well-connected structure and dense geopolymer microstructure.

The SEM images show the steel fibre surface covered with geopolymer matrix at the indicates relatively fracture surface. This good bonding between the geopolymerization product and the steel fibres, which resists pull-out failure of fibres in the FRGC sample. In the case of the PVA-FRGC mixture, PVA fibres have a coarser surface, and thickening of the fibres is observed resulting from build-up of geopolymer hydration products on the surface of the PVA fibres, indicating the strong bond between PVA fibres and the geopolymer matrix. This is in agreement with the experimental results as PVA has high post crack resistance with strain hardening behaviour. On the other hand, microscopic analysis also demonstrated that the surface of glass fibres were not roughened, and appeared relatively smooth. These fibre-matrix interface observations are clearly consistent with the mechanical behaviour of FRGC. The tensile strength and post crack behaviour are significantly improved by inclusion of PVA and steel fibres while the effect of glass fibre was less pronounced. The glass fibre showed a relatively smooth surface within the geopolymer composite, resulting in poorer anchorage in the geopolymer matrix and easy pull out during load application. In addition, the unchanged diameter of the fibres indicates negligible degradative effect of the alkaline geopolymer matrix on the steel fibres.

6.3.5 Concluding remarks.

Novel fibre reinforced geopolymer concretes with improved strain hardening performance cured at ambient temperature have been developed in this research. The geopolymer matrix was produced by using a ternary binder (fly ash, GGBS and silica fume) mix with potassium silicate alkaline activator. The effects of fibre type, volume fraction, and fibre aspect ratio on the mechanical properties of FRGC were examined. The mechanical properties of FRGC were determined by compressive strength, tensile strength and flexural strength tests. Scanning Electronic Microscopy (SEM) was also used to assess the microstructure of the examined geopolymer mixtures. The following main outcomes can be drawn from the experimental results outlined here.
• The compressive strength of the examined specimens increased by 15-25 MPa when steel fibres were added to the mix. Using PVA and glass fibres did not give the same pronounced improvement in compressive strength.

• Increasing steel fibre content and aspect ratio significantly affected the compressive strength of SFRGC. Highest compressive strength values were achieved when 3% of steel fibres with 13mm length were used, with the compressive strength value in this case found to be around 70 MPa.

- Longer straight steel fibres and higher dosage rates resulted in better mechanical performance in terms of compressive strength, tensile strength and post crack behaviour.
- The deflection hardening behaviour of a SFRGC specimen with 3% dosage of steel fibre with 13mm length gave almost twenty times higher deflection at the peak load than specimens without fibre and 4 times that of 3% of steel fibre with 6mm length.
- At the second peak load, PVA-fibre reinforced geopolymer exhibited the highest energy absorption capacity. The order of performance of different fibre types at this deflection level was as follows: PVA-fibres > ST13-fibers > glass-fibres.
- The ultimate flexural strengths of SFRGC reinforced with 13mm steel fibres was higher than that of ST6, PVA and glass fibre.
- Increasing micro steel fibre content in a hybrid system with macro steel fibres leads to enhancements in strength and deflection compared to the single macro steel fibre type. The bridging action of micro fibres leads to higher flexural strength, and can contribute towards production of better performing steel fibre-reinforced concretes. Specifically,
- Deflections of hybrid 1% V_f HE fibre with micro fibre (1% and 2% V_f) are 2 and 4 times larger than the deflection of macro 1%HE alone.
- (2) Toughness of the hybrid 1%V_F HE fibre with micro fibre (1% and 2% V_f) is 3-14 times higher for the first cracking and 3.6-5.3 times higher at second cracking than for specimens containing macro fibres alone.
- SEM analysis show that the geopolymer matrix is compacted, well connected and that the bonding between steel and PVA fibre and matrix was good. The glass fibres exhibited poorer dispersion and bonding.

In summary, this study shows that inclusion of steel and PVA fibres in the examined geopolymer matrix significantly improved the flexural and tensile strength, even without elevated temperature treatment, which makes the FRGC potentially valid for *in situ* (cast-in-place) applications. These findings warrant further research on the durability properties of this sustainable strain hardening geopolymer composite, and its suitability as a structural element. These are further explored in Chapters 7 and 8.

CHAPTER 7:

DURABILITY PERFORMANCE OF HIGH STRENGTH FIBRE REINFORCED GEOPOLYMER CONCRETE UNDER AMBIENT TEMPERATURE

7.1 Introduction.

Durability is a vital aspect of concrete character and behaviour, due to its strong relationship with the serviceability life of the structure. Concrete structures must be able to resist the mechanical action, and physical and chemical aggression that they are subjected to during their expected service life (Sahmaran et al., 2008; Ganesan et al., 2015; Shaikh and Supit, 2015a). In a mild environment, traditional OPC concrete can be a durable material. However, it has long been recognized that conventional concrete suffers from deterioration due to attack from aggressive agents such as acids, chlorides and sulphates. As clarified in the previous chapters, full replacement of OPC by geopolymer materials results in an environmentally positive impact due to usage of by-product material from various industries. Moreover, previous studies have indicated that geopolymer concrete has superior durability properties under a variety of aggressive conditions compared to OPC, because this binder system does not rely on calcium compounds and is free from C₃A (Bakharev, 2005c; Chi, 2012; Neupane, 2016). Bakharev, (2005c); (Bakharev, 2005a) found that geopolymer concrete material has a superior acid and sulphate resistance performance, while Sathia et al., (2008) examined the permeability and chemical attack of fly ash based geopolymer concrete and their results concluded that geopolymer concrete possesses excellent durability characteristics for aggressive environments. Rajamane et al., (2011) investigated the chloride ion penetration (RCPT) of geopolymer concrete (GC) and OPC concrete, and their results indicated that both GC and OPC concrete showed almost similar behaviour in respect to chloride penetration. Most of these previous studies however examined the durability properties of geopolymer cured under elevated temperature, and no study has been done to date to evaluate the durability properties of geopolymer cured under room temperature.

The development of strain hardening geopolymer concrete behaviour by incorporating randomly distributed short discrete fibres to ternary geopolymer matrix is presented in

Chapter 6. The mechanical results from this Chapter show that addition of fibre considerably improved the post cracking behavior of the geopolymer material under tension, and based on the flexural testing results, as fibre volume and aspect ratio were increased the ultimate and the residual flexural strength were significantly improved. 2% PVA and 3% steel fibre reinforced geopolymer composites exhibited multiple fine cracks and strain hardening under tension. The effect of incorporating discontinuous fibres on the durability characteristics of geopolymer materials however has not been assessed.

This chapter presents an investigation into the durability performance of plain and fibre reinforced geopolymer cured under ambient temperature, and its comparison with OPC mortar. An extensive experimental programme was conducted to examine the shrinkage characteristics and corrosion resistance of the fibre reinforced and plain geopolymer which address the research objective 3. The durability parameters considered in this study include shrinkage, corrosion resistance, resistance to chloride ions and resistance to chemical attack.

7.2 Experimental procedures.

Five different mixtures were prepared, to evaluate the effect of incorporation of variant discontinuous fibre types on the durability characteristics of the fibre reinforced geopolymer composite (Table 7.1). The optimum discontinuous fibre volume fractions from the previous chapter (Chapter 6) were 2% PVA, 3% ST13 and 1% glass. The optimum geopolymer matrix was developed in Chapter 4 by mixing (fly ash, slag and silica fume) with potassium silicate as alkaline activator. Four different tests were performed to evaluate the durability characteristics of geopolymer materials: shrinkage, corrosion resistance, chemical resistance, and rapid chloride migration (RCMT) tests. The shrinkage was assessed through measurement of dry, overlay-restrained and end-restrained shrinkage. The resistance to chemical attack was studied through use of sodium sulphate and sulphuric acid solutions, while the corrosion resistance performance was studied using induced current. The chloride permeability was assessed through measurements of non-steady state rapid chloride migration (RCMT).

Mix ID	OPC	FA/	GGBS /	SF/	Sand	K ₂ SiO ₃ /	Water/	Fibre
	$[Kg/m^3]$	Binder	Binder	Binder	$[Kg/m^3]$	Binder	Binder	$V_{\rm f}$ [%]
OPCM	650	-	-	-	1525	-	0.35	0
GPM	-	0.5	0.4	0.1	1052	0.12	0.25	0
SFRGC	-	0.5	0.4	0.1	1052	0.12	0.25	3
PVAFRGC	-	0.5	0.4	0.1	1052	0.12	0.25	2
GFRGC	-	0.5	0.4	0.1	1052	0.12	0.25	1

Table 7.1: Mixture compositions of fibre reinforced geopolymer concrete.

7.3 Results and Analysis.

7.3.1 Shrinkage characteristics of mortar and FRGC.

Shrinkage is the change in volume of concrete due to loss of water (either to internal chemical reactions or to the environment). Concrete structures are affected by four major types of shrinkage: plastic, autogenous, carbonation and drying shrinkage (Cheung and Leung, 2011; Lee *et al.*, 2014; Beushausen and Bester, 2016). Generally, shrinkage of concrete at early age is a critical parameter for determining the serviceability properties of concrete structures. Higher drying shrinkage may result in surface cracking and curling of concrete structures, leading to structural and durability problems (Neupane, 2016). Previous studies have extensively studied the shrinkage properties of conventional cementitious matrix and fibre reinforced concrete. However, the mechanism of shrinkage retardation provided by the fibres is not the same as in the case of geopolymer material, due to the difference in the matrix characteristics. In order to quantify the links between fibre type and geopolymer shrinkage performance, three different shrinkage tests (drying, overlay restrained and end restrained shrinkage) were performed on samples with different types of incorporated fibre.

7.3.1.1 Free drying Shrinkage.

Drying shrinkage is the volumetric change in the specimens as a result of movement of water from hardened concrete into the surrounding environment. Drying shrinkage occurs when concrete is exposed to a drying environment, and is the most widely recognized source of volume change in concrete (Jafarifar, 2012). A series of prismatic specimens with cross-sectional dimensions of 75 mm x 75 mm and length of 285 mm were used for measuring the free drying shrinkage properties of the geopolymer composites (Figure 7.1). Amounts of drying shrinkage were measured in accordance with (Sathia *et al.* 2008; ASTM C490/C490M 2011) using a digital gauge at 1, 3, 5,

7, 14, 21, 28, 56, 90 and 15 days. The drying shrinkage measurements were started after the final setting time and 24 hrs after casting. The specimens were stored in a room with relative humidity 42% and temperature 20 °C. In order to evaluate the general effect of fibre incorporation on the drying shrinkage performance, the drying shrinkage of plain geopolymer and OPC mortar mixtures was also determined for comparison. The average drying shrinkage results of three replicate specimens for all the examined mixtures are presented in Figure 7.2.



Figure 7.1: Free drying shrinkage set up for (a) OPCM, (b) GP mortar, (c) GFRGC, (d) PVAFRGC, and (e) SFRGC.





Figure 7.2: Dry shrinkage of plain OPC mortar and Plain geopolymer mortar up to 28 days and 120 days.

Based on the results of Figure 7.2 it is evident that the drying shrinkage strain of the plain geopolymer mortar (GPM) is very high (around 3000 microstrains) at 120 days. This is significantly higher than that of plain OPC mortar (around 1200 microstrains). This is in agreement with previous test results published by Lee *et al.*, (2014) and Collins and Sanjayan, (1999). Lee *et al.*, (2014) studied the drying shrinkage of fly ash and slag based geopolymer mortar, and their results showed that fly ash and slag based geopolymer mortar showed much higher levels of drying shrinkage than OPC mortar.

This was argued to be due to higher capillary stresses resulting from the higher mesopore volume of the fly ash and slag based geopolymer mortar compared to that of OPC mortar (Lee *et al.*, 2014). Collins and Sanjayan, (1999) noted that the drying shrinkage of ambient temperature cured slag based geopolymer concrete was higher than OPC concrete of the same grade. In contrast, Neupane, (2016) stated that fly ash and slag based geopolymer concrete had similar shrinkage characteristics to OPC concrete of the same grade.



Figure 7.3: Dry shrinkage of FRGC specimens up to 28 days and 120 days; PVAFRGC (a), GFRGC (b) and SFRGC (c).

The data points in Figure 7.3 (a-c) specify the drying shrinkage or shrinkage strain of fibre reinforced geopolymer concrete containing 2% PVA, 1% glass fibre and 3% steel fibre. The experimental results indicated that the addition of fibres leads to significant

reduction of the shrinkage strain values in all cases (i.e. PVAFRGC, GFRGC and SFRGC), and especially with the addition of steel fibres (SFRGC). Reinforcing with straight steel fibre significantly reduced the shrinkage from around 2900 microstrains to 850 microstrains, while inclusion PVA and glass fibre reduced the shrinkage strain to around 1600 microstrains and 1400 microstrains, respectively at 120 days. This reduction is attributed to the physical restraint provided by the presence of the fibres in the geopolymer matrix, which is in agreement with previous studies of fibre reinforcement conventional concrete (Zhang and Li, 2001; Li *et al.*, 2006). Li *et al.*, (2006), reported that the reduction of the drying shrinkage strain is observed even when using a low volume fraction of the fibres. Duran Atiş *et al.*, (2009) supported this finding and also reported that the use of steel fibre restrained the movements of microlevel in fly ash and OPC based concrete by bridging and stitching the fine cracks.

It can be seen from the Figures (7.2 and 7.3) that the rate of shrinkage was high at early ages, up to 28 days, and the rate gradually decreased after this age. The increase in the drying shrinkage of plain geopolymer up to 28 days is from 1500 to 2700 microstrain, while the shrinkage increases from 2700 to 2900 microstrain between 28 days and 120 days. In the case of fibre reinforced geopolymer this trend is more pronounced, as around 85% of the total drying shrinkage was achieved at 28 days compared with drying shrinkage values at 120 days.

7.3.1.2 Restrained shrinkage behaviour.

Cracking that occurs in concrete due to the restraint of shrinkage is the main concern with respect to durability. Shrinkage of concrete is resisted by external resistance from the underlying and/or end boundary against shortening, or by internal restraint against non-uniform volumetric change (Kwon and Surendra, 2008). The concrete restrained shrinkage behaviour is relatively complicated as it depends on many parameters, for instance the drying shrinkage magnitude and rate, development of strength and modulus of elasticity, creep and tensile relaxation, tensile stress, geometry of specimens and degree of restraint (Yoo *et al.*, 2015). The restrained shortening at early age may stress the concrete in excess of its early tensile strength (ACI 360R, 1992), and it is well recognized that shrinkage movements lead to serious cracking (microcracks and macro-cracks) in concrete (Hughes, 2003). The restrained shrinkage of conventional mortar, concrete and fibre reinforced concrete has been examined in

previous studies (Li *et al.*, 2006; Beushausen and Chilwesa, 2013; Nam *et al.*, 2016) and there is no widely accepted relationship between the measured shrinkage and restrained shrinkage, which represent the actual shrinkage of a repair material (Abbasnia *et al.*, 2005).

To facilitate the mix design of fibre reinforced geopolymer shotcrete for practical application, it is crucial to design a laboratory set up that can evaluate the effectiveness of FRGC mixtures against shrinkage cracking. Several methods have been proposed in the past for the evaluation of the effect of restrained shrinkage. Nevertheless, an alternative approach of performing shrinkage tests representing the two possible scenarios when considering the fracture response from an overlay repair technique (overlay-restrained and end restrained tests) is used in this study (Carlswärd, 2006). Figure 7.4a showed the well bonded overlay to the underlying substrate, while the debonding part of the overlay material to the substrate which represent the end-restrained shrinkage (Figure 7.4b).



Figure 7.4: (a) Evenly distributed restraint in case of full bond to the substrate and (b) end restraint over debonded area (Carlswärd, 2006).

7.3.1.2.1 Overlay-restrained shrinkage.

Casting a new layer of concrete over an existing substrate can be used for strengthening and structural repair, i.e. where the overlay is stress loaded and performs a structural role, or for non-structural repairs to protect exposed reinforcing steel or to replace damaged concrete (Bissonnette *et al.*, 2013). The differential shrinkage between the substrate and the new layer material is based on the concept that the overlay is subjected to thermal movement and shrinkage, while the substrate's movements are minimal or have completed. Bonded overlay deformation is therefore

restrained at the interface with the substrate, resulting in tensile stresses and causing overlay cracking (Beushausen and Alexander, 2006; Dittmer and Beushausen, 2014). Plain geopolymer mortar and FRGC overlays were cast on concrete substrate beams in order to evaluate the differential shrinkage behaviour of FRGC in structural applications, and compared with conventional mortar and concrete overlay material. Substrates with dimensions of 500 mm length, 100 mm width and 100 mm depth were cast and left to mature for six months prior to application of a 50 mm thick overlay (Figure 7.5). The binder of the concrete substrate was ordinary Portland cement (OPC) with a cement amount of 380 Kg/m³ and water to cement ratio of 0.5, with 37 MPa compressive strength. To facilitate bonding between overlay and substrate, an air chipping hammer was used for substrate surface roughening, as shown in Figure 7.6. All composite beams, the concrete surface roughened to a depth of 2-3 mm, representing as "a well-roughened" fib Bulletin 55, (2010) and concrete surface texture characterised by following Sand Patch Test procedure ASTM E965-96, (2006). Before emplacing the overlay material, compressed air was used to thoroughly clean the substrate beam surfaces, which were then pre-wetted to a saturated dry surface before application of 50 mm thick overlays. After placing the overlay materials, the fresh repair materials were vibrated by a vibrating table to remove air bubbles particularly at the interface, which are deleterious to interfacial bonding. Then, the overlay materials were covered with plastic sheeting and cured for 24 hours. After demoulding, the layered specimens were moved into a room under ambient curing conditions of (20-23 °C). Under these conditions, the shrinkage of the repair materials developed and caused cracking of the overlay material.



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Figure 7.5: Composite beam (concrete substrate with a layer of overlay); Side view (a) and Top view (b).



Figure 7.6: (a) untreated surface and (b) roughened surface.

As shown in Figure 7.5, the composite beams consisted of the overlay material bonded on top of the concrete substrate. The additional layer was reinforced with two reinforcement bars of diameter 10 mm with a layer cover of 25 mm in order to represent the common overlay repair technique which is applied in the repair and strengthening techniques used in this research study (Chapter 8). The overlays were designed as out-stretched strips with a width of 100 mm, a length of 500 mm and a depth of 50 mm. As can be seen in Figure 7.7 there were a total of twelve composite beams with two identical beams for each material overlay; plain geopolymer (PGM), OPC mortar (OPCM), normal strength concrete (NSC), SFRGC, PVAFRGC, and GFRGC.

7.3.1.2.1.1 Strain development of the overlay material.

To be able to track the deformation of the overlay shrinkage, pairs of Demec points were attached to the surface of each strip across a length of 100 mm by using Epoxy Adhesive. Locations of the Demec points are illustrated in Figures (7.5b and 7.7) and the specimens were unsealed on all surfaces. A strain dial gauge with a gauge length of 100 mm was used for the measurements. The Demec Gauge was manually placed between two succeeding points, attached at a distance of 100 mm, and the measuring accuracy was 1 micrometre. Shrinkage strains were measured several times during the first month and over a total period of 120 days. All results presented in Figure 7.8 and Figure 7.9 are an average of the restrained shrinkage results distribution of specimens along the overlay material length.



Figure 7.7: Composite overlay-substrate specimens.



Figure 7.8: Strain distribution at different ages in the overlay strip surface of plain and FRGC material; NSC (a), OPCM (b), PG (c), GFRGC (d), PVAFRGC (e), and SFRGC (f).



Figure 7.9: Longitudinal strain distribution at the top overlay strip surface of plain and FRGC material; NSC (a), OPCM (b), PGM (c), GFRGC (d), PVAFRGC (e), and SFRGC (f).

The restrain shrinkage development of the overlay material start from immediately after demoulding up to 120 days age, as shown in Figure 7.8 and Figure 7.9. The shrinkage strain at the top surface responded differently based on the shrinkage

resistance performance of the overlay material. Based on the experimental results, the overlay surface contracted up to ca. 28 days age, and then maintained an almost constant level for 28-60 days. A slight increase in contraction was then measured over the remaining period at the top of the overlay layer. The 28 days restrained shrinkage of the PVAFRGC overlay was higher than SFRGC and less than GFRGC overlay material, which is in agreement with the drying shrinkage results.

Quite similar shrinkage strain distribution response over the material overlay length was noticed for all FRGC materials. The uncrack shrinkage strain recorded at the free edge was much higher than that value at the centre line of the overlay material. This is related to the stress state developing at the free ends due to the transfer of normal stresses from the overlay to the substrate (Carlswärd, 2006). This behaviour could be explained due to the restrained shrinkage of overlay FRGC material reduced by presence internal reinforcing rebar and prevent the freely deformed in any direction by the volumetric change in the overlay material (Yoo *et al.*, 2015). Yoo *et al.*, (2015) clarified the conceptual view of the restrained shrinkage behaviour of concrete with an internal reinforcing bar (Figure 7.10). If no creep effect is assumed, the shrinkage strain can be divided into two classes: elastic restraint strain by rebar ε_e and elastic rebar stain $\varepsilon_{e,r}$ (c). However, in reality, owing to the tensile creep of concrete ε_{cr} , the strain obtained in the rebar (or in concrete) by shrinkage decreases to ε_r (d). Therefore, the free shrinkage strain ε_{sh} is equal to the summation of ε_e , ε_{cr} , and ε_r (Yoo *et al.*, 2015).



Figure 7.10: Conceptual view of restrained shrinkage behaviour of concrete with internal rebar (Yoo *et al.*, 2015).

7.3.1.2.1.2 Cracking in overlay material.

The differential shrinkage between the overlay material and concrete substrate led to cracking of the overlay materials and interface debonding in these composite beams. A summary of the results of the overlay cracking at the end of the testing period is given in Table 7.2, where the time to cracking and the free shrinkage strain at the corresponding time are given. The crack width was calculated directly from the strain measurements following the procedure explained in Figure 7.11. The term uncracked is the mean value of the measured strains in un-cracked parts of the specimen, i.e. obtained over the three measuring lengths where no crack was detected. L_m is the measuring distance of 100 mm and L_{m1} is the distance after cracking.



 $\Delta L_m = L_{m1} - L_m = w + \varepsilon_{uncracked} \cdot L_m$ $w = \Delta L_m - \varepsilon_{uncracked} \cdot L_m$

Figure 7.11: Principles for calculation of crack widths. Lm is the measuring length, Lm1 is the distance after cracking and uncracked is the strain in uncracked parts (Carlswärd, 2006).

Visual inspection showed that cracking occurred for plain geopolymer overlay almost immediately after demoulding the composite beam. Several cracks with widths equal to or less than 0.3 mm were observed for both composite beams of plain geopolymer overlay. This is clear when studying the strain distributions at selected ages measured on the top surface of the outermost overlay strips of plain geopolymer mortar (GPM), as shown in Figure 7.11 and Figure 7.12. The first crack in PVAFRGC was observed after 7 days with multiple cracking behaviour, while first cracking was observed after 14 days in the conventional mortar overlay material. Only one of the two composite beam specimens with GFRGC overlay material cracked after 11 days. However, there were no cracks observed in the surface of SFRGC and NSC composite beams.

120 days, the number of cracks increased in the PVAFRGC overlay. These had very small crack widths, and in many cases, were hairline cracks, but were present in larger numbers than in the GFRGC and OPCM overlays. After 120 days, an average of eight cracks with width ranging from 30 μ m to 60 μ m were formed in the surface of the PVAFRGC overlay, while one crack with a width of 40 μ m was formed in the surface of the GFRGC overlay material.

A general conclusion that may be drawn from the results that the conventional steel bar provide significant contribution to crack width limitation. The maximum crack width for all examined overlay material expect plain geopolymer mortar were 0.3 mm or below (Table 7.2 and Figure 7.12). The addition of fibre prolonged the time until a crack developed: the PVAFRGC and GFRGC overlay materials did not crack until after 7 to 11 days at corresponding free shrinkage of 1000 μ m/m, while specimens reinforced with a 3% volume fraction of steel fibre did not crack at all over the 120 day period of testing. This can be explained as follows: fibre was added to the concrete mixture randomly, and so only some fibres were distributed parallel to the direction of shrinkage strain. When the matrix was subjected to tensile stresses induced by shrinkage, the fibres oriented parallel to the shrinkage strain restrain the shrinkage strain by shear along the fibre-matrix interface, and thus reduce it. Therefore, the higher the fibre contents used in the concrete the more reduction in shrinkage strain is expected. In addition, fibres make the composite stronger and stiffer. Moreover, the presence of coarse aggregate considerably improves the shrinkage performance as presented in the normal strength concrete overlay. Schematic top views of the experimental overlay materials after 120 days are presented in Figure 7.13.

Overlay	Time to	Cracking	No. of
material	cracking width		cracks
		[mm]	
OPCM	28 days	0.05 mm	2
PG	2 days	0.05 mm-0.3	11
PVAFRGC	7 days	0.03 mm-0.06	7
GFRGC	11 days	0.05	1
SFRGC			
NSC			

Table 7.2: Results from the overlay restrained shrinkage test.



Figure 7.12: Cracking in the overlay surface of plain and FRGC material; OPCM (a), NSC (b), GFRGC (c), PVAFRGC (d), SFRGC (e), and PG (f).



Figure 7.13: Surface crack pattern of different layered repair systems based on OPCM (a), NSC (b), GFRGC (c), PVAFRGC (d), SFRGC (e), and PG (f) after 120 days.

7.3.1.2.2 End-restrained shrinkage.

The end restrained shrinkage testing configuration used is illustrated in Figure 7.14. In the present investigation, a box steel beam with polished upper face was employed as the restraining substrate, giving a smooth surface to ensure that restraint would be developed at the end of the specimens only. L-shaped steel supports of 40 x 40 mm with a thickness 4 mm were secured to the restrained substrate by bolts at distance of 500 mm. Square angle steel supports were then fastened along the sides before the concrete was placed. Just before casting, the smooth surface of the upper face of the steel substrate was oiled and 150 X 40 X 8 mm thick steel plate was loosely positioned directly at the oiled and smooth surface of the upper flange to allow for easy removal after hardening. Restraint was achieved through three 10 mm threaded bars at each Lshaped support extending approximately 75 mm into the concrete. After casting, the surface was covered by plastic sheeting to allow for a curing period of 24 hours. The test was initiated by removing the cover, the sides and steel platen to avoid base friction. Demec points for deformation measurements were fixed to the concrete surface by using epoxy glue at a distance of 100 mm starting from the square angle support. Thus, a total of four measuring distances were obtained along the concrete specimens.



Figure 7.14: Set-up of end restrained shrinkage test (all dimensions in mm).

To demonstrate the applicability of the proposed set-up, shrinkage cracking experiments were performed with plain geopolymer and conventional mortar and fibre reinforced geopolymer composite containing PVA and steel fibre. For each mixture, the test rigs for end restrained shrinkage and free shrinkage specimens were placed alongside each other to evaluate the unrestrained deformation under similar conditions (Figure 7.15).



Figure 7.15: Test rigs of end restrained shrinkage specimens and specimens for measuring the free shrinkage.

7.3.1.2.3 Strain development in end restrained specimens.

The strain development at the top of specimens prepared using ordinary Portland cement mortar (OPCM) is shown in Figure 7.16a while the corresponding results of geopolymer (plain and fibre reinforced geopolymer containing PVA and steel fibre) are shown in Figure 7.16 (b- e). Free shrinkage results at the upper face of the identical specimens of all the examined mixtures are also shown in Figure 7.16.



Figure 7.16: Strain development along the restrained specimens over curing time, with corresponding measured free shrinkage (identical specimen) shown at the top; OPCM (a), PG (b), PVAFRGC (c) and SFRGC (d).

As expected, inclusion of fibres significantly reduced the restrained shrinkage strain development over the curing time. From the results in Figure 7.16, when PVA and steel fibre is added to mixes, the mean of the un-cracked restrained shrinkage reduced by about 50% and 75% respectively. It can be seen that the growth of FRGC shrinkage strain was considerably slower than that in the plain concrete specimen. These results indicate the high effectiveness of fibres in reducing shrinkage of fibre reinforced geopolymer composite, due to these fibres bridging the crack and transferring load from one side to the other, thus preserving the stress in the concrete to some extent. It is also clear that crack strain considerably increased while the strain in the un-cracked parts of the plain and PVAFRGC specimens approach the curve representing free shrinkage. This is because no stresses were transferred through the crack, and the uncracked parts were completely un-loaded.

7.3.1.3 Cracking in end restrained specimens.

The strain distribution along the specimen's length and over the curing time are presented in Figure 7.17. Table 7.3 shows the summary of the end restrained shrinkage results including number of cracks, crack width and time to cracking.



Figure 7.17: Longitudinal strain distribution of a restrained specimen at the specimen's top surface over curing time; OPCM (a), PGM (b), PVAFRGC (c) and SFRGC (d).

Material	Time to	No.	Crack width	
type	cracking	of		
	[Days]	cracks	[mm]	
OPCM	7	1	0.15	
PG	2	1	0.2	
PVAFRGC	7	1	< 0.03	
SFRGC	No crack			

Table 7.3: Summary of results from the restrained shrinkage tests.

The crack width for the cracked specimens are determined directly from the measured strain as discussed earlier in section 7.3.1.2.1.2. The cracks occurred within the first week after test initiation for all the examined mixtures apart from the SFRGC mixture, as here the reduction in shrinkage provided by the presence of steel fibres was sufficient to prevent cracking entirely (by bridging and stitching fine cracks). Table 7.3 shows that cracking occurred after 2 days and 7 days of demolding for PG and OPCM respectively, and the crack width increased with curing time. Inclusion of PVA fibres considerably reduced the cracking width by 5 times and multiple cracking did not occur, in contrast to results obtained for overlay shrinkage (Figure 7.18).



Figure 7.18: Major cracks observed on plain geopolymer and OPC mortar on end restrained shrinkage specimens.

7.3.1.3.1 General conclusions of restrained shrinkage.

The effect of discontinuous fibres on shrinkage-induced cracking in thin geopolymer concrete overlays has been assessed. Overlay restrained shrinkage and end-restrained shrinkage tests were used to evaluate the crack response of overlays. It was found that

crack distributions and crack widths in the overlay material were different with different types of fibre used. In general, however inclusion of steel, PVA and glass fibres effectively reduced the differential shrinkage in thin bonded overlays.

In terms of test method suitability, the end restrained test is useful for assessing the crack sensitivity of different fibres or variant material compositions, as similar restraint conditions are provided for all tests. However, the test set-up must be adjusted somewhat in order to ensure consistency of the results and to enable evaluations of FRGC restrained shrinkage. On the other hand, the overlay restrained shrinkage is more precise from an overlay perspective as it records the restraint situation more accurately. However, the quality of the overlay bond to the substrate complicates the assessment of the fibre type on the shrinkage resistance (Carlswärd 2006).

7.3.2 Corrosion resistance

The corrosion of reinforcing steel bars is one of the major causes of concrete structure deterioration, resulting in the reduction of concrete service life. Resistance of reinforcement rebar against corrosion depends upon the chemical composition of the surrounding medium (Torres-Acosta *et al.*, 2007; Sahmaran *et al.*, 2008; Xie and Hu, 2013). Generally, steel rebar embedded in new concrete is protected against corrosion by the high alkalinity of concrete pore waters (pH~13), which passivates the steel surface. However, concrete deterioration still occurs as a result of premature reinforcement corrosion, particularly where structures are located in the coastal marine environment.



Figure 7.19: Mechanism of corrosion of steel in concrete (Etman, 2012).

Corrosion of steel takes place in concrete due to a number of initiating causes that expose the bars to moisture and oxygen either by carbonation or chloride intrusion (Chen and Zhou, 2010; Etman, 2012; Wu *et al.*, 2014). During the process of cement hydration, a thin protective alkaline passive film is formed around the rebar. A corrosion process is initiated when this protective film is broken. The steel surface depassivates when the chloride concentration reaches threshold levels on the reinforcing bar surface or when the pH of the concrete cover drops below critical levels due to carbonation. The corrosion procedure that occurs in concrete is electrochemical in nature, and very similar to a battery. Corrosion will result in the flow of electrons between anodic and cathodic sites on the rebar (Figure 7.19). When corrosion is initiated, active corrosion leads to structural failure by reducing the cross-sectional area of the reinforcement bar and by causing a volumetric expansion of the rust around the steel bars against the surrounding concrete (Figure 7.20) (Daily, 1999; Broomfield J. P., 2003). As a result, longitudinal corrosion cracks form in the concrete along the corroding steel bar due to the corrosion-induced tensile stress, and rehabilitation is commonly required at this stage of structural deterioration (Choi *et al.*, 2006).



Figure 7.20: Stages of corrosion-induced cracking of the concrete (Daily, 1999).

Globally, the repair and replacement costs of concrete structures in old infrastructure are measured in billions of dollars (Sahmaran *et al.*, 2008). To reduce this tremendous cost, the service life of reinforced concrete infrastructure needs to be substantially improved by using an adequate corrosion control material in reinforced concrete exposed to severe environmental conditions (Yodsudjai, 2014). Geopolymer is receiving increasing attention as an alternative to Portland cement because of its high strength, durability and low environmental impact (Lee and Lee, 2013). There is very limited research however examining the corrosion resistance of geopolymer concrete. In this thesis, fibre reinforced geopolymer concrete has been developed to achieve high strength and deflection hardening performance following curing under ambient

temperature. This study examines the performance of fibre reinforced geopolymer composite beams under a corrosive environment (i.e. addressing thesis objective 3). Experimental test results are used to validate the failure resistance of FRGC, determine the mass loss of steel bar and to provide the technical basis for using FRGC in extending the infrastructure service life. Electrochemical techniques applied for inducing accelerated corrosion of steel in concrete are applied.

7.3.2.1 Accelerated reinforcement corrosion by Induced current test.

Corrosion of steel reinforcement in concrete is a slow process (Ahmad, 2009), taking a considerable length of time for initiation and then progression of steel bar corrosion under corrosion exposure conditions due to the protective nature of concrete. It is difficult to obtain measurable reinforcement corrosion over the limited time periods typically available for laboratory-based research studies. For this reason, an accelerated corrosion testing technique was employed to accelerate the corrosion and shorten the test period (Etman, 2012; Shaikh and Supit, 2015a), in order to compare the corrosion performance of conventional mortar and FRGC.

Conventional mortar and FRGC prism specimens with dimensions 75 mm x 75 mm x 285 mm were used after 28 days curing. Deformed steel bars of 12 mm diameter and 500 mm in length were fixed in the centre of the specimens after cleaning and accurate weighing. The steel bar has an effective cover of 25 mm at the bottom. The prism specimens were partially immersed in a 5% sodium chloride solution. The exposed steel bar was then connected to the positive terminal of a DC power source while the negative terminal was connected to stainless steel plates placed near the specimen in the solution. Figure 7.21 shows a schematic representation of the accelerated corrosion test, and the experimental set up is presented in Figure 7.22. The corrosion process was initiated by applying a constant current for specific periods. Such corrosion periods mimic those of many years in real structures (Sahmaran *et al.*, 2008). The current required for variant desired degrees of induced corrosion for different material is tabulated in the Table 7.4.



Figure 7.21: Schematic diagram of the accelerated corrosion test used.



Figure 7.22: Experimental set up: DC power supply (a) and Corrosion tanks used for accelerated corrosion test (b).

corrosion rate (ρ)= 8%	corrosion rate (ρ)= 12%		
Initial steel bar weight= 500 gram	Initial steel bar weight= 500 gram		
Proposed time $(T) = 5 x 24 x 60 x 60$	Proposed time $(T) = 10 x 24 x 60 x 60$		
= 432000 second	= 864000 second		
$Mass \ loss = Wi - Wf = Wi \ x \ \rho$	$Mass \ loss = Wi - Wf = Wi \ x \ \rho$		
$= 500 \ x \ 0.08 = 40 \ gram$	$= 500 \ x \ 0.12 = 60 \ gram$		
(Wi - Wf)xF	(Wi - Wf)xF		
$\frac{1}{\pi x D x L x W x T}$	$\frac{1}{\pi x D x L x W x T}$		
$Icorr = \frac{(40 \times 96487)}{\pi \times 12 \times 50 \times 27.027 \times 422000}$	$Icorr = \frac{(60 \times 96487)}{\pi \times 1.2 \times 50 \times 27.027 \times 864000}$		
$\pi x 1.2 x 30 x 27.927 x 432000$	$\pi \times 1.2 \times 50 \times 27.927 \times 864000$		
$= 0.00168 \frac{mp}{cm^2}$	$= 0.001273 \frac{mp}{cm^2}$		
Icorr - Icurrent	Icorr - Icurrent		
$\frac{1}{\pi x D x L}$	$\frac{1}{\pi x D x L}$		
$I \ current = 0.0168 \ x \ 3.1415 \ x \ 1.2 \ x \ 50$	$I \ current = 0.0127 \ x \ 3.1415 \ x \ 1.2 \ x \ 50$		
= 0.319 Amp	= 0.24 Amp		
Use 0.3 Amp for up to 5 days	Use 0.3 Amp for up to 10 days		

Table 7.4: Time calculation for different degree of corrosion.

For the flexural strength test, prism specimens with a 75 x 75 mm cross section, 285 mm in length, and a support span length of 160 mm were tested in a closed-loop machine after having been exposed to different degrees of accelerated corrosion (Figure 7.23). In each case, two replicate specimens were loaded until failure to determine their load-deflection curves and ultimate flexural load capacity. Three control prisms of each mixture without accelerated corrosion exposure were also tested after 28 days curing as references (Table 7.5 and Figure 7.24).

Mixture ID	Accelerated time (days)	No. of specimens
		3
OPCM	5	2
	10	2
		3
SFRGC	5	2
	10	2
PVAFRGC		3
	5	2
	10	2

Table 7.5: Mixtures examined at different degrees of accelerated corrosion.



Figure 7.23: Flexural test setup.



Figure 7.24: Prisms after flexural test (a) OPCM, (b) PVAFRGC, and (c) SFRGC.

7.3.2.2 Effect of corrosion on the Load-Deflection relationship.

The experimental flexural strength was determined to investigate the efficiency of local protective materials on the corrosion of steel reinforcement. A control (without corrosion) and corroded beams after inducing different degrees of accelerated corrosion were tested under centre point bending to determine their flexural load deflection curves and ultimate flexural loads. A centre point flexural test was applied to the face of the beam where corrosion induced cracks were observed. The load deflection relationship of the conventional mortar and FRGC mixtures after 0, 5 and 10 days exposure to corrosion are illustrated in Figure 7.25, Figure 7.26, Figure 7.27 and Figure 7.28, respectively. Average flexural load deflection for the beam specimens were obtained by combining the results of two or three specimens tested in each case.



Figure 7.25: Load-deflection curves of prism specimens; SFRGC (a), PVAFRGC (b) and OPCM (c).



Figure 7.26: Load-deflection curves of corroded prism specimens after exposure to accelerated corrosion for 5 days; SFRGC (a), PVAFRGC (b) and OPCM (c).



Figure 7.27: Load-deflection curves of corroded prism specimens after exposure to accelerated corrosion for 10 days; SFRGC (a), PVAFRGC (b) and OPCM (c).



Figure 7.28: Comparison of load-deflection curves of not exposure, 5 days exposure and 10 days exposure for SFRGC prisms (a), PVAFRGC prisms (b), and OPC prisms.

Mixture ID	Accelerated time [days]	Maximum flexural load [kN]	Flexural load reduction [%]	Failure type
OPCM		21		Shear failure
	5	12	43	Bond failure
	10	8	62	Bond failure
SFRGC		36		Flexural failure
	5	31.5	13	Flexural failure
	10	27.7	23	Flexural failure
PVAFRGC		27.5		Flexural failure
	5	24.5	11	Flexural failure
	10	20.7	25	Flexural failure

Table 7.6: Ultimate flexural load at different degrees of accelerated corrosion, for ordinary Portland cement and fibre-reinforced geopolymer specimens.

Load-deflection relationships of the conventional mortar and FRGC specimens before and after different accelerated corrosion periods (corroded, x days) are shown in Figures 7.25-7.28 and summarized in Table 7.6. The maximum flexural load of the control (without corrosion) fibre reinforced geopolymer composite prism was considerably higher than that of conventional mortar. The maximum flexural loads were 21 kN, 36 kN, and 27.5 kN for OPCM, SFRGC, and PVAFRGC, respectively. The corrosion of the steel reinforcement had a clear impact on the flexural load deflection performance of the specimen prisms. These impacts varied according to the different protective materials used (Table 7.6). Exposure of specimen prisms to current for 5 days decreased the ultimate load to 8 KN, 31.5 and 24.5 KN for OPCM, SFRGC and PVAFRGC mixtures, respectively (Figure 7.26). The ultimate load capacity of the conventional mortar prisms decreased sharply as corrosion progressed. However, the influence of accelerated corrosion testing of up to 10 days on the load-deflection curves of the FRGC specimens is relatively minor. The ultimate flexural load of the SFRGC, PVAFRGC and OPCM prisms decreased after 10 days corrosion by 23%, 15% and 80% for SFRGC, PVAFRGC and OPCM respectively compared to the respective ultimate load values of the control (without corrosion) mixture (Figure 7.27). This reduction in the ultimate load is a result of the loss of the cross-sectional area of steel reinforcement and, moreover, to the presence of multiple micro-cracking in the case of FRGC, and localized macro cracking for conventional mortar. Deflection at the ultimate load of the corroded PVAFRGC and conventional mortar specimen prisms was higher than the deflection of the control (without corrosion) beams. The SFRGC corroded specimens had deflection less than deflection of the corroded beams.

Visual observation showed that PVAFRGC specimens exhibited multiple cracking behaviour, with small crack spacing and tight crack widths. Flexural failure in the SFRGC and PVAFRGC mixtures occurred when the fibre bridging strength at one of the microcracks was reached, resulting in localized deformation at this section (Figure 7.29). FRGC beams without shear reinforcement displayed significant shear resistance, because of their strain-hardening and multiple-cracking behaviour under tensile loads (Sahmaran *et al.*, 2008). Conventional mortar failed by shearing due to low strength characteristics. When the beams were exposed to corrosion, the type of failure of the corroded FRGC specimens remained the same as in non-corroded specimens. Corrosion of the steel reinforcement changed the failure mode in the conventional mortar however as the corroded beams failed by bond splitting due to the reduction in the bond between the steel reinforcement and the matrix. The corrosion-induced longitudinal cracking along the reinforcing bar in the mortar specimens leads to a loss of frictional mechanical bonding.



Figure 7.29: Mode of failure of control and corroded prisms under centre point bending test, before and after the accelerated corrosion test: (a) OPCM, (b) SFRGC and (c) PVAFRGC.

It can be observed from the load deflection curves that as the corrosion degree increased the area under the load deflection curve decreased. The area under the load deflection curve represents the absorbed energy and toughness. The slope of the loaddeflection curve represents the stiffness of the beams, and it is apparent that the slope decreases with an increasing degree of reinforcement corrosion. Thus, increasing the degree of corrosion also influences the ductility and brittleness behaviour of the material. To facilitate the comparison between the test results for conventional mortar and FRGC specimens, the area under the load deflection curve up to the maximum load was examined. The OPCM prisms showed a decrease in area under the load deflection curve of 70% and 85% after exposure to corrosion for 5 days and 10 days respectively, compared with the area under the load deflection curve of the control beams.

The ductility reduction in FRGC prisms was less pronounced after accelerated corrosion, with PVAFRGC prisms showing a decrease in area under the load deflection curve of 12% and 19% after 5 days and 10 days applied current, and SFRGC prisms showing a decrease of 18% and 40% compared with the corresponding values of the control beams. This difference between FRGC and conventional mortar is due to the former's ability to resist corrosion expansion during induced corrosion due to the presence of fibres, leading to smaller and less connected microcracks. Microcrack widths of less than 0.1 mm (Figure 7.31) were observed on the surface of FRGC prisms, which slow down further chloride penetration and reduce the rate of corrosion propagation. Tuutti, (1982) proposed that crack widths of less than 0.1 to 0.3 mm did not affect the corrosion rate of the reinforcing steel (Figure 7.31).



Figure 7.30: Crack distribution of corroded prisms after flexural testing: localized crack on SFRGC specimen (a,) and multiple cracks distribution (b).


Figure 7.31: Major cracking on the corroded prisms after 10 days induced current.

7.3.2.3 Effect of corrosion on the mass loss of the steel reinforcement.

To evaluate the effect of different protective materials on the mass loss of the reinforcing bar after exposure to different degrees of accelerated corrosion, the reinforcing bar was extracted from the concrete by breaking the specimens. The reinforcing steel for each specimen was cleaned with a stiff metal brush to ensure that the steel bar was free from any adhering corrosion products. The reinforcing bar was then weighed and the percentage mass loss was computed using Eq. (7.1). The actual mass of corroded products per unit surface area is measured according to Eq. (7.2).

corrosion mass =
$$\frac{[\text{initial mass} - \text{final mass}]}{\text{initial mass}} \times 100$$
(7-1)

$$Mac = \frac{[initial mass - final mass]}{\pi DL}$$
(7-2)

Where Mac is actual mass of rust per unit surface area of the bar (g/cm^2) , the initial and final mass refer to the mass of the reinforcing bars (g) before and after corrosion, D is diameter of the rebar (cm), L is length of the rebar sample (cm).



Figure 7.32: Corrosion status of reinforcing steel bar embedded in concrete; OPCM (a), PVAFRGC (b), and SFRGC (c).

Figure 7.32 shows the actual corrosion of steel in the conventional ordinary Portland cement mortar and SFRGC and PVAFRGC specimens after the accelerated corrosion test. Significant corrosion can be seen on the surface of the rebars in OPCM specimens. In contrast, the steel rebar taken from FRGC samples containing PVA and steel fibres suffered less corrosion as indicated by the reduction in the weight loss of the rebar (in Table 7.7 and Figure 7.33).

Mixture ID	Duration	Initial mass	Final mass	Actual mass	Percentage of
	[day]	[g]	[g]	$loss [g/cm^2]$	mass loss [%]
OPCM	5	441	375	0.324	15
	10	438	337	0.492	23
SFRGC	5	445	425	0.098	4.5
	10	447	398	0.241	11
PVAFRGC	5	439	424	0.075	3.5
	10	443	412	0.152	7

Table 7.7: Corrosion mass-loss measurement of rebar from different types of material.



Figure 7.33: Mass loss versus corrosion exposure time for conventional mortar, SFRGC and PVAFRGC corrosion specimens: total mass loss rate (%).

The percentage of steel mass loss in the plain geopolymer mortar and FRGC specimens over different accelerated corrosion exposures is presented in Table 7.7 and Figure 7.33. The average percentage mass loss of reinforcing steel bars embedded in the conventional mortar specimens was 15% and 23% after 5 days and 10 days of accelerated corrosion exposure, respectively. On the other hand, there was very small mass loss in the reinforcing bars embedded in the FRGC specimens. The average percentage of mass loss in PVAFRGC and SFRGC specimens was 3.5% and 4.5% after 5 days exposure, and 7% and 11% after 10 days of accelerated corrosion exposure.

This difference in the mass loss of the reinforcing bars with different protection materials occurs because of the crack width and distribution after accelerated corrosion exposure. OPCM specimens formed wide longitudinal cracks along the specimens causing higher accessibility and direct penetration and contact of the chloride ions with the reinforcement. The crack widths were sufficient to allow easy migration of corrosion products, confirmed by visual inspection of rust on the crack surfaces of the severely damaged specimens. In contrast, SFRGC and PVAFRGC specimens exhibited tight crack widths which prevent the movements of the corrosion products that serve as a shield against chloride ion migration toward the steel bar (Sahmaran *et al.*, 2008; Masmoudi and Bouaziz, 2016). Thus, the accelerated test conditions indicate that the use of FRGC reduced the corrosion of rebar significantly when compared with a conventional mortar cover.

7.3.3 Non-steady State Chloride Migration (RCMT).

The durability properties of fibre reinforced geopolymer concrete are rarely found in the literature, and most previous studies focus on plain geopolymer concrete. In this thesis, a rapid chloride migration (RCM) test was used for conventional mortar, plain geopolymer mortar, and FRGC to investigate resistance against chloride penetration. The RCM test was carried out according to the Nordic test method (NT Build 492, 1999).

For each mixture, 100 x 200 mm cylinder specimen was cast and cured as described in Chapter 5. After 90 days curing, three cylindrical specimens with a diameter of 100 mm and a thickness of 50 mm were cut to produce a 50 ± 2 mm thick slice from each specimen, taken from the central portion of the cylinder (Figure 7.34). After being surface dried, specimens was placed in a vacuum container for 3 hours at a pressure of 10-50 mbar, prior to being saturated with calcium hydroxide (Ca(OH)₂) solution (by dissolving an excess of calcium hydroxide in distilled or de-ionised water) for 18±2 hours as shown in in Figure 7.35.



Figure 7.34: A specimen cut using a special dry cutting blade (a), and cut specimens used for RCM tests (b).



Figure 7.35: Specimens in vacuum saturation apparatus.

After limewater (Ca(OH)₂) saturation, the specimens were put in a rubber sleeve and secured by two clamps to prohibit water ingress along the edge of the specimens, as shown in Figure 7.36. The sleeve above the specimen was filled with 300 ml anolyte solution (0.3 M NaOH), and then the whole sleeve was immersed in the catholyte solution of 10% NaCl. The experimental set-up and schematic diagram explaining the specimen placement in the apparatus are shown in Figure 7.37 and Figure 7.38, respectively. An electrical potential of 30V was applied axially across the specimen, forcing the chloride ions to migrate from outside to inside the specimen. Based on the initial current, the test applied voltage and the test duration is selected (as shown in Table 7.8) (Jóźwiak-Niedźwiedzka, 2009). For instance, if the initial current is between 60 and 90 mA, the test voltage is reduced to 25 V DC and the test duration is 24 hours (Table 7.8).



Figure 7.36: Specimen sealed with rubber sleeve and stainless-steel clamps.



Figure 7.37: RCM test set-up.



Figure 7.38: Schematic diagram of the set-up of the apparatus for the Nordic Test Method (NT Build 492, 1999).

After 24 hrs exposure to chloride solution, the rubber sleeve and the clamps were removed and the 3 specimens split into two pieces by applying load using compressive crushing machine. A 0.1 M silver nitrate (AgNO₃) solution was sprayed onto the freshly split surfaces. AgNO₃ reacts with NaCl and produces AgCl. AgCl appears white and indicates the chloride penetration depth. After around 15 minutes of spraying AgNO₃ solution, the penetration depth was measured at 7 locations from the centre to both edges at intervals of 10 mm according NT Bulid 492 standard. The average penetration depth was determined by averaging the measured values from each specimen. The NORDTest (NT Build 492, 1999) method data is expressed as a non-steady migration coefficient (D_{nssm}, m²/s). The chloride coefficient calculated using the following equation:

$$D_{nssm} = \frac{0.0239(273+T)L}{(U-2)t} [x_d - 0.0238 \sqrt{\frac{(273+T)Lx_d}{U-2}}$$
(7-3)

Where, D_{nssm} is the apparent non-steady state migration coefficient (m²/s x10⁻¹²), *U* is the applied potential (volts), *L* is the specimen thickness (mm), *T* is the average value of the initial and final temperatures in the anolyte solution (°C), x_d is the penetration depth (m) and *t* is the test duration (sec).

	-		
Initial current	Applied voltage U	Possible new	Test duration
I_{30V}	(after adjustment)	initial current Io	t
(with 30 V)			
[mA]	[V]	[mA]	[hour]
$I_0 < 5$	60	$I_0 < 20$	96
$5 \leq I_0 < 10$		$10 \leq I_0 < 20$	48
$10 \leq I_0 < 15$		$20 \leq I_0 < 30$	24
$15 \leq I_0 < 20$	50	$25 \leq I_0 < 35$	
$20 \leq I_0 < 30$	40	$25 \leq I_0 < 40$	
$30 \leq I_0 < 40$	35	$35 \leq I_0 < 50$	
$40 \leq I_0 < 60$	30	$40 \leq I_0 < 60$	
$60 \leq I_0 < 90$	25	$50 \leq I_0 < 75$	
$90 \leq I_0 < 120$	20	$60 \leq I_0 < 80$	
$120 \le I_0 < 180$	15	$60 \leq I_0 < 90$	
$180 \le I_0 < 360$	10	$60 \leq I_0 < 120$	
$I_0 \geq 360$		$I_0 \ge 120$	6

Table 7.8: NT Build 492 (NordTest) method – Test voltage and recommended duration (NT Build 492, 1999).

7.3.3.1 Non-steady State coefficient.

For samples subjected to the accelerated NT Build 492 procedure, silver nitrate (AgNO₃) is used to reveal chloride penetration depths. The chloride penetration depths were measured on three cylindrical samples for each mixture are presented in Figure 7.39.



Figure 7.39: Chloride penetration depth results (n=3).

The conventional mortar has the deepest chloride penetration depth (x_d) of around 24.3 mm, while the plain geopolymer mortar shows lower chloride permeability compared to the conventional mortar tested here, at around 11.3 mm (Figure 7.40 and Table 7.9). These low depths of penetration are attributed to the dense C–N–A–S–H binding gels formed in geopolymer materials, which hinder chloride penetration (Ismail *et al.*, 2013). The binding gel chemistry affects pore structure in terms of geometry and radius, as well as total pore volume. The presence of slag powder in the geopolymer mortar produces high contents of geopolymer gel, and reduces porosity, giving lower penetration of chloride (Lee and Lee, 2013). The presence of fibres in the geopolymer slightly increases the chloride penetration depth to 13.5 mm and 13.2 mm for PVAFRGC and SFRGC, respectively. The chloride penetration of SFRGC mixture was not measured in this test due to the higher current which passed through these specimens, which significantly affected the test results. The chloride migration coefficient of the conventional mortar, plain geopolymer and fibre reinforced geopolymer are presented in Table 7.9 and Figure 7.41.



Figure 7.40: Test of the chloride resistance for specimens; OPCM (a), GPM (b), PVAFRGC (c) and GFRGC (d).

MIX ID	N.O.S	L	Т	U	t	x_d	D _{nssm}
OPCM	1	50.25	21.85	25	24	25.26	14.25
	2	47.35	21.85	25	24	24.71	13.17
	3	45.39	21.85	25	24	23.02	11.75
PG	1	52.15	22.65	30	24	11.68	5.36
	2	48.7	22.6	30	24	10.79	4.62
	3	49.33	22.4	30	24	11.33	4.93
GFRGC	1	46.3	10.35	30	24	13.13	5.26
	2	49.75	10.35	30	24	13.02	5.56
	3	49.925	10.35	30	24	14.35	6.20
PVAFRGC	1	50.5	10.2	25	24	12.41	6.39
	2	51.2	10.25	25	24	13.41	7.05
	3	50.75	10.3	25	24	13.78	7.20

Table 7.9: Test results details.



Figure 7.41:Non-steady state chloride migration (NT BUILD-492) coefficient.

The conventional mortar gave the highest non-steady state migration coefficient, while the plain geopolymer mortar gave the lowest (Figure 7.41, showing D_{nssm} , m²/s). The non-steady-state migration coefficient was reduced from $13x10^{-12}$ m²/s for ordinary Portland cement mortar to 5 x10⁻¹² m²/s for plain geopolymer cement. Inclusion of glass fibres and PVA fibres slightly increase the chloride coefficient to 5.7x 10⁻¹² m²/s and 6.9x10⁻¹² m²/s for GFRGC and PVAFRGC mixtures, respectively. This is possibly due to an increase in the volume of interfacial porosity at fibre–matrix interfaces, which provides easier transport paths for chloride (Banthia *et al.*, 2014).

The chloride migration coefficient under non-steady-state (D_{nssm}) can be used for classifying concrete resistance to chloride penetration, according to the criteria presented in Table 7.10 (Jóźwiak-Niedźwiedzka, 2009). Based on these assumptions and the measured chloride coefficient values, the plain geopolymer and both PVAFRGC and GFRGC mixtures can be classified as having good resistance to chloride penetration, while the resistance of conventional mortar can be classified as acceptable.

· /	
Chloride migration coefficient	Resistance to chloride
D [m ² /s]	penetration
$< 2 \times 10^{-12}$	very good
$2 - 8 \times 10^{-12}$	good
$8 - 16 imes 10^{-12}$	acceptable
$> 16 \times 10^{-12}$	unacceptable

Table 7.10: Estimation of the concrete resistance to chloride ion penetration (Jóźwiak-Niedźwiedzka, 2009).

7.3.4 Resistance to chemical attack.

The chemical resistance of plain geopolymer and fibre reinforced geopolymer concrete was assessed by measuring the residual compressive strength and change in mass after exposure to sulphate solutions and acidic solutions. After 28 days of casting, the 50 mm cube specimens were oven dried at 105 °C and weighed. Out of twenty-eight cube specimens of each mixture, four specimens without exposure were used as control specimens for estimating the loss in compressive strength and initial weight loss. For each mixture, eight specimens from each mix were immersed in sulphuric acid and another eight cubic specimens from the same batch immersed in sodium

sulphate solution, and tested after 3 and 6 months (Figure 7.42). For comparison, for every period of exposure, a set of specimens from the same batch was also cast, kept in ambient temperature and covered with plastic sheet until testing. All specimens were cured under room temperature.

Sodium sulphate (Na₂SO₄) solution with 3% concertation was used as the standard exposure solution (Wallah and Rangan, 2006). The specimens were immersed in the sodium sulphate solution in a container after noting their initial weights (Figure 7.43a). To test the acid resistance of geopolymer material, following previous studies, the specimens were immersed in 3% sulphuric acid (H₂SO₄) solution in a container for up to 6 months, and the solution stirred every week (Figure 7.43b).



Figure 7.42: Schematic diagram of the set-up of specimens soaked in sealed containers.



Figure 7.43: Specimens soaked in solution: (a) Sodium Sulphate solution and (b) Sulphuric Acid solution.

On the day of the test, the specimens were removed from the solution container and wiped clean. Specimens were oven dried at 105 °C and weighed before testing. The

chemical resistance was evaluated based on visual appearance, change in mass, and change in compressive strength after chemical exposure over three months and six months periods.

7.3.4.1 Effect of chemical attack on visual appearance.

The visual appearances of the examined cubic specimens after different exposures to sodium sulphate and sulphuric acid are shown in Figure 7.44. There was no visible change in the specimens after soaking in sodium sulphate solution for up to 6 months (Figure 7.44). There was no sign of surface erosion, spalling or cracking on the cubic specimens. On the other hand, erosion and colour change of the specimen's surfaces were observed in conventional mortar and geopolymer specimens after exposure to sulfuric acid solution (Figure 7.44).



Figure 7.44: Concrete specimens after exposure for six months to (left) Sodium Sulphate and (right) Sulphuric acid.

7.3.4.2 Effect of chemical attack on the compressive strength.

7.3.4.2.1 Effect of Sodium sulphate exposure on the compressive strength.

A series of test were performed to examine changes in compressive strength after sodium sulphate solution exposure for plain geopolymer and fibre reinforced geopolymer specimens cured under ambient temperature, and compared to conventional mortar. Test results for specimens soaked in sodium sulphate solution for up to 3 and 6 months are presented in Figure 7.45 and Figure 7.46, respectively.



Figure 7.45: Compressive strength of specimens after 3 months of exposure to sodium sulphate solution.



Figure 7.46: Compressive strength of geopolymer after 6 months of exposure to sodium sulphate solution.

The test data in Figure 7.45 and Figure 7.46 showed that exposure of geopolymer mortar and fibre reinforced geopolymer to 3% sodium sulphate solution caused very little change in the compressive strength. On the other hand, the ordinary Portland cement deteriorates by sulphate attack. Results are summarized in Table 7.11.

Mixture ID	Difference	ce of comp nth compre (without o	ressive stre essive stren exposure)	Difference or compressive st respective period strength (witho	f exposure rength to the d compressive out exposure)	
	3 mo	onths	6 mo	nths	3 months	6 months
	Exposed	Non-	Exposed	Non-	Exposed	Exposure
		exposed		exposed		
OPCM	-8	18	-15	23	-21	-31
PG	11	7	16	11	4	4
PVAFRGC	7	5	20	14	2	6
SFRGC	17	13	38	33	3	4
GFRGC	14	7	19	16	7	2

Table 7.11: Summary of the test results of sodium sulphate resistance.

It can be seen from Table 7.11 that the compressive strength slightly increased after exposure to sodium sulphate. At 3 months of sodium sulphate exposure, the compressive strength of plain geopolymer mortar increased by 11% and 4% relative to the compressive strength of non- exposed geopolymer at 1 month and 3 months, respectively. Fibre reinforced geopolymer specimens showed a similar trend to the plain geopolymer, for example SFRGC compressive strength increased by 17% and 3% compared to the compressive strength value of the control mixture at 1 month and 3 months, respectively. In contrast, the compressive strength of the OPCM mixture reduced after exposure to sodium sulphate solution to 8% and 21% of the compressive strength of the control mixture after 1 month and 3 months, respectively. After 6 months exposure to sodium sulphate, the loss in the compressive strength of OPCM specimens was around 31% compared with the control (without exposure) specimens. In contrast, the compressive strength of plain and fibre reinforced geopolymer specimens.

This difference in behaviour is driven by the different chemistry of the geopolymerization products and the Portland cement hydration products. The deterioration of the ordinary Portland cement mortar after sulphate attack is most likely due to the reaction of sulphate ions with portlandite to form gypsum, or due to the reaction of sulphate ions with calcium alumina hydrate to form calcium sulfoaluminate or ettringite (Wallah and Rangan, 2006). The formation of expensive gypsum and ettringite due to sulphate attack causes expansion, cracking and spalling in the conventional mortar (Ganesan *et al.*, 2015). Moreover, the sulphate attack on

the Portland cement leads to loss of strength in the concrete due to the loss of cohesion in the hydrated cement paste and of adhesion between it and the sand/ aggregate particles (Neville, 2000). On the other hand, geopolymer don't contain Ca(OH)₂ and mono-sulphoaluminate in the matrix to cause expansion (although the formation of lower amounts of ettringite and gypsum could have occurred due to the presence of calcium in slag) (Singh *et al.*, 2015a). These results demonstrate the excellent resistance of the plain geopolymer mortar and fibre reinforced geopolymer cured under room temperature to sulphate attack.

7.3.4.2.2 Effect of sulfuric acid on the compressive strength.

Sulfuric acid is one type of acid solution that is frequently used to simulate acid attack in the infrastructure concrete systems. There are no standard procedures for acid resistance testing, and so the type and concentration of the acid solution to which specimens have been exposed have varied (Wallah and Rangan, 2006). To evaluate the change in compressive strength after acid solution attack, specimens were soaked in 3% sulphuric acid solution. The test results for ambient-temperature cured plain geopolymer mortar and fibre reinforced geopolymer immersed in sulphuric acid for 3 months and 6 months are presented in Figure 7.47 and Figure 7.48, respectively, and compared to control specimens (without exposure).



Figure 7.47: Compressive strength of geopolymer after 3 months of exposure to sulphuric acid solution.



Figure 7.48: Compressive strength of geopolymer after 6 months of exposure to sulphuric acid solution.

Figure 7.47 and Figure 7.48 show the degradation in the compressive strength of conventional mortar, geopolymer mortar and FRGC due to sulphuric acid solution attack. The summarized test data showing compressive strength after acid exposure as a percentage of the control (without exposure) after 1 month, 3 months and 6 months are presented in Table 7.12.

Table 7.12: Test results of Sulphuric acid resistance.

Mixture ID	Difference of compressive strength to 1 month compressive strength (without exposure)				Difference of exposure compressive strength to the respective period compressive strength (without exposure)		
	3 mo	onths	6 mo	onths	3 months	6 months	
	Exposed	Non-	Exposed	Non-	Exposed	Exposed	
		exposed		exposed			
OPCM	-18	18	-35	23	-30	-47	
PG	-2	7	2	11	-8	-8	
PVAFRGC	2	5	1	14	-2	-11	
SFRGC	8	13	20	33	-4	-10	
GFRGC	2	7	5	16	-4	-10	

The reduction in the compressive strength of geopolymer due to sulfuric acid exposure depends on the period of exposure. Following 3 months of acid exposure, the compressive strength of the conventional Portland cement mortar reduced to 18 % and 30 % of the compressive strength of the control (non-exposure) mortar after 1 month

and 3 months. The degradation of the geopolymer was much less pronounced than that observed in the conventional Portland cement mortar. After 3 months of sulphuric acid attack, the compressive strength of plain geopolymer decrease by 2 % and 8 % compare with the compressive strength of control (non-exposed) mortar after 1 month and 3 months, respectively. The degradation in compressive strength increased as the period of exposure to the acid solution increased, from 3 months to 6 months. The reduction in the compressive strength of PVAFRGC, SFRGC and GFRGC mixtures increased from 2 %, 4 % and 4 % after 3 months exposure to 11 %, 10 % and 10 % after 6 months, compared with the respective compressive strength values of the reference (without exposure) samples at 3 months and 6 months, respectively. Maximum strength loss suffered by the FRGC mixture was 10% whereas for OPCM it was around 47%. The results also indicate that the fly ash and slag based geopolymer specimens cured under ambient temperature showed superior resistance to sulphuric acid solution compared to the conventional mortar.

Loss in compressive strength of geopolymeric materials exposed to sulphuric acid solution has also been reported in previous studies (Bakharev, 2005c; Song *et al.*, 2005). The reduction in the compressive strength after sulphuric acidic solution attack is related to depolymerisation of aluminosilicate polymers in the acidic media and the formation of zeolites (Bakharev, 2005a). However, the geopolymeric materials perform much better resisting acid attack than Portland cement, which is in agreement with previous studies (Song *et al.*, 2005; Wallah and Rangan, 2006). This could be due to the lower calcium content of the source material: of the geopolymer binder materials used in this study (40% slag, 10% silica fume and 50% fly ash) only slag contains around 40% CaO, while 65% CaO is present in Portland cement mortar. Since the calcium content in the geopolymer used was very low for the geopolymer specimens exposed to H₂SO₄, the resulting formation of calcium sulphate was much less significant, therefore, the geopolymer material performed better than the Portland cement mixtures in the acidic environment.

7.3.4.3 Effect of chemical attack on the mass loss.

• Effect of Sodium sulphate attack on the mass loss.

After the specific testing periods, the specimens were removed and their dry weights were noted. Test results of the change in mass of the specimens immersed in sodium sulphate solution after 3 months and 6 months as a percentage of the mass before exposure are presented in Figure 7.49.



Figure 7.49: Change in mass of specimens soaked in sodium sulphate solution at 3 months and 6 months.

It can be seen that there was no reduction in the mass of the specimens, as confirmed by the visual inspection of the specimens in Figure 7.49. There was a slight increase in the mass of specimen's due to the absorption of the exposed liquid. The increase in mass of plain geopolymer and fibre reinforced geopolymer specimens immersed in sodium sulphate solution was approximately 1.5% after three months of exposure. Increasing the exposure time does not increase the mass apart from in the PVAFRGC mixture as the mass increased by 1.23% at 3 months to 2.2% at 6 months. The % mass loss of the OPCM specimens increased with exposure time from 3.25 to 5.6%.

• Effect of Sulphuric acid solution attack on the mass loss.

To determine change in mass after sulphuric acid attack, the immersed specimens in sulphuric acid solution were removed, dried and weighed after 3 months and 6 months. Data on the change in mass of conventional mortar, plain geopolymer mortar and

FRGC specimens after 3 months and 6 months as a percentage of the mass of the specimens before exposure are presented in Figure 7.50.



Figure 7.50: Change in mass of specimens soaked in sulphuric acidic solution at 3 months and 6 months.

The mass loss of plain geopolymer was about 1.7% and 3% after 3 months and 6 months exposure to sulphuric acid solution. The same trend was observed for fibre reinforced geopolymer, for instance the reduction in mass of SFRGC was about 1.3% and 2.9% after exposure for 3 months and 6 months, respectively. These mass losses are significantly smaller than those of conventional concrete. The reduction in mass of ordinary Portland cement was around 11.3% and 16.2% after acidic attack for 3 months and 6 months, respectively. The outcome of this study is in agreement with previous research studies (Gourley and Johnson, 2005; Ganesan *et al.*, 2015), which confirm the superior performance of fly ash based geopolymer concrete in acidic environments compared to Portland cement concrete. For example, Song *et al.*, (2005) noted in their experiments that the mass loss of fly ash based geopolymer was less than 3% after 56 days of exposure to 10% sulphuric acid solution while the conventional concrete lost 41% of its mass after 28 days of exposure.

7.4 Concluding remarks.

The durability performance of plain geopolymer mortar and fibre reinforced geopolymer concrete cured under ambient temperature have been examined in this chapter and compared with the performance of conventional mortar. The optimum geopolymer matrix, fibre types and volume fractions identified in previous chapters were examined. The present chapter has presented the results and discussion of an experimental study conducted to investigate the shrinkage characteristics, corrosion resistance, non-steady state chloride migration and chemical resistance. The following main conclusions can be drawn from the experimental results outlined here.

- The drying shrinkage of geopolymer mortar was higher than that of the OPC mortar, whereas inclusion of discontinuous fibres (2%PVA fibre, 1% glass fibre and 3% steel fibre) in the geopolymer mortar considerably reduced the drying shrinkage by 45%, 53% and 70%, respectively.
- Throughout experimental investigation using overlay restrained and endrestrained tests, inclusion of discontinuous fibre was found to result in better cracking behaviour in terms of crack width, distribution and time before cracking compared to the plain geopolymer mortar.
- Induced corrosion caused large cracks along the length of the conventional mortar specimen, and the crack width increased with time as corrosion activity progressed. On the other hand, the cracks in SFRGC and PVAFRGC specimens were very small and remained almost constant with time as corrosion activity progressed.
- Corrosion of reinforced conventional mortar specimens resulted in a significant reduction of stiffness and ultimate load capacity. In contrast, the effect of induced current corrosion had only a small effect on the flexural load deflection capacity of SFRGC and PVAFRGC specimens.
- Longitudinal cracks resulting from the expansion of the corrosion products change the failure mode of the reinforced conventional mortar under flexural test. On the other hand, the corrosion of the steel bar in FRGC specimens does not change the type of failure in FRGC beams.
- Geopolymer mortar and FRGC specimens demonstrate higher performance than conventional mortar against chloride ingress, according to non-steady state chloride migration (NordTest NT Build 492).
- The experimental results indicate that the geopolymer mortar and FRGC specimens cured under ambient temperature have an excellent resistance to sulfuric acid attack, as there is no substantial change in the mass and the

compressive strength increased by 2-4% after exposure up to 6 months, while the compressive strength of conventional mortar specimens decrease by 31% and 5.6% mass loss.

• The sulfuric acid resistance of geopolymer mortar and FRGC specimens is significantly better than that of conventional mortar as indicated in experimental results. Exposure to 3% sulfuric acid solution for up to 6 months reduces the compressive strength of geopolymer mortar specimens by 11% and FRGC specimens by 8%, while the compressive strength of the conventional mortar reduced by 47%.

The observed superior corrosion and chemical resistance of FRGC compared with conventional mortar in term of corrosion propagation time, tight crack width, lower mass loss, and higher flexural load, is attributable to the high tensile strain capacity, strain hardening, and multiple-cracking behaviours of FRGC. Overall, these experimental findings suggest that the FRGC material could be used as a repair material to increase the service life of old structures and as a strengthening material to improve the carrying capacity. This is further explored in Chapter 8.

CHAPTER 8:

STRENGTHENING AND REPAIR OF EXISTING REINFORCED CONCRETE (RC) BEAMS FOLLOWING EXPOSURE TO SEVERE ENVIROMENTAL CONDITIONS

8.1 Introduction.

Over the last few years interest in the repair of reinforced concrete (RC) structures has increased, as the premature degradation of RC structures exposed to severe environmental conditions and excessive mechanical loading has become an increasingly serious problem. The strengthening of existing structures can also improve structural response to seismic loading (Maheri *et al.*, 2012; Lampropoulos *et al.*, 2016). In addition, there are many infrastructural elements, such as bridges or tunnels, which have to be rehabilitated to overcome the social and economic costs associated with the demolition and the reconstruction of new structures (Martinola *et al.*, 2010). Some estimates indicate that, globally, in 2010 the expenditure for maintenance and repair work represented about 85% of the total expenditure in the construction field (Grantham *et al.*, 2009; Mourad and Shannag, 2012). Therefore, the development of long-lasting and effective repair/strengthening methods can greatly reduce these maintenance requirements, improve safety and increase the service life of concrete structures.

Reinforced concrete (RC) structures are durable because of steel reinforcements in concrete is prevented from corroding by a tight passive film formed on its surface due to high alkalinity of the surrounding concrete (Kobayashi *et al.*, 2010). However, when chloride ions penetrate the cover concrete and reach the steel surface, the passive film becomes destructed and outbreak of steel corrosion. In the presence of sufficient oxygen, chloride, moisture and if an electrical circuit is formed, the steel starts to corrode resulting in decrease the cross-sectional area of a steel bar, spalling of the cover concrete, which can lead to deterioration of the RC structure performance (Ahmad, 2009; Rajamane *et al.*, 2011; Kobayashi and Rokugo, 2013). Chloride attack is one of the most serious causes of damage to RC structures, because of its diffusion and the rapidness of deterioration process. Thus, once such deterioration occurs in a

reinforced concrete structure, it must be repaired appropriately in order to avoid further degradations and to get their lost functions recovered.

There are numerous research projects and publications focusing on the repair of deteriorating old structures. However, most of this research uses traditional strengthening techniques based on externally bonded steel plates, reinforced concrete jacketing (CEB Bulletin d'Information 162, 1983; Cheon and MacAlevey, 2000), and use of externally bonded Fibre Reinforced Polymer (FRP) (Dawood and Rizkalla, 2010; Yi *et al.*, 2010; Yun and Wu, 2011; Attari *et al.*, 2012). All of these techniques can be used with favourable results but have some limitations. In particular, the use of externally glued FRPs as well as steel plates could have issues for fire resistance. The use of reinforced concrete jacketing systems need to apply concrete layers with thickness larger than 60–70mm as the presence of reinforcing bar requires a minimum concrete cover (Martinola *et al.*, 2010).

Fibre reinforced cementitious composites (FRCC) have been developed and extensively researched over the last two decades (Shaikh and Hosan, 2016). Generally, the addition of fibres to a concrete mix considerably enhances many of the mechanical properties of concrete such as flexural, impact, tensile and abrasion strength, and post cracking behaviour (Uygunoğlu, 2008; Al-Majidi et al., 2017b). One of the most promising areas of application of this material is in the repair of concrete structures. Recently, novel techniques using fibre reinforced concrete (FRC) layers or jackets have been proposed to improve the performance of existing structural members (Martinola et al., 2010; Hussein et al., 2012; Lampropoulos et al., 2016). Simultaneously, fibre reinforced geopolymer concretes (FRGC) have emerged as novel engineering materials with the potential to form a substantial element of an environmentally sustainable construction and building products industry(Al-Majidi et al., 2017a). In the current research, fibre reinforced geopolymer composites with higher ductility and strain hardening behaviour have been developed (Chapter 6). Moreover, the FRGC material developed in this research showed superior durability characteristics in term of sulphuric acid attack, corrosion resistance and chloride penetration (Chapter 7). The efficiency of FRGC material in structural behaviour has been rarely studied, and there are (to date) no published studies on the evaluation of FRGC as repair material with comparison to other traditional strengthening methods such as the use of Reinforced Concrete (RC) layers and jackets. This is despite

potential for FRGC use in repair and strengthening of existing structures to improve both durability and loading capacity.

The aim of this Chapter is to examine the efficiency of the newly developed fibre reinforced geopolymer concrete cured under ambient temperature for strengthening of RC beams, and to investigate improvements in bending capacity compared to a reference beam. The work presented in this chapter addresses thesis objectives 4 and 5. The mechanical performance of reinforced concrete beams retrofitted/ strengthened with FRGC was investigated using four-point bending tests. Large scale beams strengthened with additional FRGC layers reinforced with steel bars have been examined. Polyvinyl fibre reinforced geopolymer concrete (PVAFRGC) and steel fibre reinforced geopolymer concrete (SFRGC) materials were used as strengthening and repair materials for the protection of the steel bars of the new layer, and subsequent improvement of the flexural strength of existing beams. Also, an additional investigation has been conducted in beams where part of the concrete cover at various depths has been replaced by PVAFRGC. In all the examined cases respective beams made from conventional concrete have been examined in order to evaluate the effectiveness of the proposed technique. Accelerated corrosion studies were also undertaken using the induced current technique by applying a nominal 300 μ A/cm² constant anodic current for approximately 30-90 days.

8.2 Experimental Procedures.

8.2.1 Reinforced concrete beam geometry, preparation and material properties.

In total 26 reinforced concrete beams were constructed for this study, four as a reference beam (without treatment), eight for repair applications with different repair thickness, while the rest of the beams (n=16) were cast in similar dimensions to the reference beam and then strengthened with FRGC and normal strength concrete (NSC) layers/jackets (Table 8.1). For each examined case, two identical RC beams were examined due to it was found to be reasonably managed and handled according to the available facilities in the lab and following the procedure has been done by previous studies (Kobayashi *et al.*, 2010; Mourad and Shannag, 2012). The test beams were classified into two series, the beams in series 1 are the control specimens (non-corroded beams) while the beams in series 2 are the RC beams exposed to corrosion (Table 8.1). Figure 8.1 shows the dimensions of beams where the length is 1400 mm,

breadth is 200 mm and the width is 100 mm. The reinforcement consisted of two deform bars with a diameter of 10 mm ($2\Phi10$) made of steel with a characteristic yielding stress value of 530 MPa in the tensile side. Stirrups of 8 mm deformed bars diameter ($\Phi8$) were used in the shear span at an interval of 90 mm with a measured yield strength stress value of 350 MPa and spacing 90 mm. The ultimate strengths, for the $\Phi8$ and $\Phi10$ bars were 414 MPa and 640 MPa, respectively (Table 8.2 and Figure 8.2). The specimens were fabricated from ordinary Portland cement. Coarse aggregates having particle size <10 mm as well as fine aggregates of 5 mm were used to prepare the ordinary concrete. The strengthening material properties of NSC, PVAFGC and SFRGC have already been explained in Chapter 7 and are listed in Table 8.2. During casting, concrete cubes with dimension of 100 mm were sampled and tested for compressive strength, which at the time of structural testing was equal to 32 MPa. The experimental casting procedure of the initial beams are presented in Figure 8.3.

Table 8.1: Number of specimens for each parameter.

	RC member	No. of specimens		
		Corroded	Non-Corroded	
Repair	Reference RC beam	2	2	
technique	PVAFRGC (25mm cover thickness)	2	2	
	PVAFRGC (50mm twice cover thickness)	2	2	
Strengthening	PVAFRGC (50mm Addition layer)	2	2	
technique	SFRGC (50mm Addition layer)	2	2	
	NSC (50mm Addition layer)	2	2	
	PVAFRGC (3 Side Jacketing)	-	2	



Figure 8.1: Reference beam-geometry and reinforcement details (all dimensions in mm).

Material	Compressive	Tensile	Elastic	Yield	Ultimate
	strength	strength	modulus	stress	stress
	[MPa]	[MPa]	[GPa]	[MPa]	[MPa]
NSC	43	-	31.2	-	-
SFRGC	70	3.8	25	-	-
PVAFRGC	46	3.5	24	-	-
$\Phi 8$ bar	-	-	202	350	414
Φ10 bar	-	-	202	530	650

Table 8.2: Material mechanical properties.



Figure 8.2: Stress-strain curves of steel reinforcement bar Φ 8 (a) and Φ 10 (b).



Figure 8.3: Experimental procedure/set-up of RC beams; placement of the reinforcement cages (a) and RC beams after casting (b).

8.2.2 Repair and strengthening of reinforced concrete beam.

Three basic configurations were examined to evaluate the effectiveness of applying FRGC materials in the strengthening of existing concrete structures, which are: replacing the damaged concrete for protection purposes with FRGC material (Repair method); and casting an additional layer, and three side jacketing, to increase the flexural capacity resistance (Strengthening methods).

8.2.2.1 Repair Method.

In this method, the concrete side under tension was replaced with a PVAFRGC layer. The repaired thicknesses of 25 mm and 50 mm (Figure 8.4) correspond to the waterproofing layer of structural elements (Safdar *et al.*, 2016). The RC beams with ordinary concrete were cast in a wooden mould up to the desired depth (Figure 8.5), demoulded after 2 days and left to mature up to 3 months. Since the concrete cover of deteriorated RC beams must be removed before the surface coating or the repair

material is applied on actual deteriorated structures, in this study FRGC was overlaid on RC beams without initial deterioration of the reinforcement bars. The exposed steel reinforcements were cleaned using a steel brush (Figure 8.6a), and an air chipping hammer was used for roughening the concrete surfaces (Figure 8.6b) followed by washing out by a high-pressure water-jet to clean the surface of dust and to remove the weak layer of dry cement paste and loose aggregate (Figure 8.7). After 3 months, a layer of PVAFRGC was cast directly on the concrete beam surface by overturning the RC beam upside down (Figure 8.8). Since the curing of PVAFRGC was carried out at ambient temperature and humidity, a plastic sheet was placed on the surface in order to limit water evaporation. After demoulding, the PVAFRGC surface was kept under wet condition by spray curing for the first 10 days, to avoid cracking resulting from differential shrinkage.



Figure 8.4: Schematic diagram of repair technique.



Figure 8.5: Casting the substrate RC beams for repair purposes.



Figure 8.6: Preparation of the concrete surface: Roughening the concrete surface (a), and cleaning the reinforcement steel (b).



Figure 8.7: The initial beams before treatment (a) and after cleaning (b).



Figure 8.8: Initial beams ready for casting the repair layer at 25 mm thickness (a) and 50 mm layer thickness (b).

8.2.2.2 Strengthening Methods.

Fourteen identical beams were cast and left to mature for 3 months for strengthening purposes (Figure 8.9). At an age of 90 days, the surfaces of the beams were roughened and cleaned as discussed above in repair methods (Figure 8.10). For all of the strengthened beams, the concrete surface was roughened to a depth of 2–3 mm by using air chipping hammer, representing "a well-roughened" concrete surface texture These definitions are in agreement with those found in fib Bulletin 55, (2010). There are different procedure to describe concrete surface texture (Santos P, 2010). The Sand Patch Test according to ASTM E965-96, (2006) was used in this study. Strengthening was performed by adding a new concrete layer with 50 mm thickness on the beams' tensional side (Figure 8.11). Four identical beams were strengthened with additional layers of each strengthening material (PVAFRGC, SFRGC) and compared with a conventional strengthening technique using normal strength concrete. The cubic concrete compressive strengths of the normal strength concrete determined on the day of testing were equal to 43 MPa. In addition, two beams were strengthened by 3 side jacketing in order to study the best possible technique efficiency of the material (Figure 8.12). In the case of the strengthening layer on the tensile side, the additional layer was reinforced with $2\Phi 10$ steel with a concrete cover of 25 mm (Figure 8.11a), while the 3-side jacketing was unreinforced (Figure 8.12a).







Figure 8.10: Prepared concrete surfaces: roughened concrete surface for strengthening layer application (a), roughened concrete surface for 3 Side jacketing (b), and high-pressure water application (c).



Figure 8.11: RC beams before overlay application (a) and after casting of the strengthening layer (b).



Figure 8.12: Prepare the mould before applying 3 side layers (a) and after casting the 3-side strengthening layer (b).

8.2.3 Accelerated corrosion test by impressed current technique.

Since the objective of this research is to evaluate the efficiency of applying FRGC to retrofit reinforced structures against severe environment conditions, an induced accelerated corrosion test was employed to simulate the corrosion of steel reinforcement in concrete. A schematic of the accelerated corrosion test setup is presented in Figure 8.13. Twelve RC beams were corroded by an accelerated corrosion system, and twelve specimens were un-corroded as the reference beams (Table 8.3). In this corrosion system, RC beams were immersed in a 5% sodium chloride solution to accelerate the corrosion. The corrosion process was accelerated by impressing a constant current of 300 mA for a given time between the reinforcement bar (anode) and a copper mesh (cathode) at the bottom surface of the container connected to negative terminal of the DC power supply, as shown in Figure 8.14. The anodic connection to the reinforcement bar were different with different retrofitting techniques. For repaired beams, the reinforcement bars of the initial beams were connected to the anodic of the DC power while, 2 deformed steel bars of 10 mm diameter in the additional layer of the strengthened beams connected to the anodic of the DC power. The visual appearance of the beam was inspected regularly, and the test was stopped when localized cracks from corrosion were detected.



Figure 8.13: Accelerated corrosion system: Schematic diagram of corrosion pool (a); circuit of accelerated corrosion for strengthening layer (b), 25 mm repair layer (c) and 50 mm repair layer (d).



Figure 8.14: Set-up for accelerating reinforcement corrosion in RC beam specimen; Power supply (a), and specimens under accelerated corrosion process (b).

Retrofitting techniques	Time of corrosion exposure [days]	Description	N.O.S	Specimen designation
Reference		Initial RC beam	2	Ref
beam	90	Initial RC beam	2	Ref-corr
		PVAFRGC (25mm cover thickness)	2	PVAFRGC-R25
Repair	90	PVAFRGC (25mm cover thickness)	2	PVAFRGC-R25-corr
technique Strengthening technique		PVAFRGC (50mm twice cover thickness)	2	PVAFRGC-R50
	90	PVAFRGC (50mm twice cover thickness)	2	PVAFRGC-R50-corr
		NSC strengthening layer	2	NSC-S50
	30	NSC strengthening layer	2	NSC-S50-corr
		PVAFRGC strengthening layer	2	PVAFRGC-S50
	30	PVAFRGC strengthening layer	2	PVAFRGC-S50-corr
		SFRGFC strengthening layer	2	SFRGC-S50
	30	SFRGFC strengthening layer	2	SFRGC-S50-corr
		3 side jacketing	2	PVAFRGC-3SJ

Table 8.3: Test RC beams details.

8.2.3.1 Time required for different percentages of corrosion.

In order to determine the corrosion level of the RC beams, the mass loss rate of reinforcing bars due to applied current over a given time was regarded as representing the corrosion rate, which can be measured according to the assumption that the corrosion state is uniformly distributed within the tensile steel material (Shaikh and Supit, 2015b). In order to achieve the required mass loss of steel, the corrosion time was estimated using Faraday's law, which is expressed by the following equation:

$$M_{th} = \frac{WI_{app}T}{F} \tag{8-1}$$

 M_{th} = theoretical mass of rust per unit surface area of the bar (g/cm²), W= equivalent weight of steel which is taken as the ratio of atomic weight of iron to the valency of iron (27.925 g), I_{app} = applied current density (Amp/cm²), T = duration of induced corrosion (s), F = Faraday's constant (96,487 Amp-sec).

The actual mass of rust per unit surface area was determined gravimetrically in accordance with ASTM Committee G-1, (2011), using reinforcement bars taken from the concrete following breaking of the specimens after the accelerated corrosion test (Eq. 8.2). The degree of induced corrosion is also expressed in terms of the percentage weight loss (ρ) calculated as expressed in Eq. 8.3.

$$M_{ac} = \frac{Wi - W_f}{\pi DL} \tag{8-2}$$

$$\rho = \frac{Wi - W_f}{Wi} \times 100 \tag{8-3}$$

Where M_{ac} = actual mass of rust per unit surface area of the bar (g/cm²), Wi = initial weight of the bar before corrosion (g), W_f =weight after corrosion (g) for a given duration of induced corrosion (T), D = diameter of the rebar (cm), L = length of the rebar sample (cm).

The equivalent corrosion current density (I_{corr}) can be estimated by equating Eq. 8.1 and Eq. 8.2, assuming that the theoretical and actual mass of rust are equal (i.e., $I_{app} = I_{corr}$), as

$$I_{corr} = \frac{\left(W_i - W_f\right) x F}{\pi D L W T}$$
8-4)

The current required for varying desired degrees of induced corrosion for different retrofitting techniques is tabulated in Table 8.4.

Table 8.4: Time calculation for different degree of corrosion.

For strengthening applications	For repair applications
For corrosion rate (ρ)= 12%	For corrosion rate (ρ)= 10%
Initial steel bar weight	Initial steel bar weight
$= 8.05 x 3.1415 x (0.5)^2 x 270$	$= 8.05 x 3.1415 x [((0.5)^2 x (270 x 2))$
= 1700 gram	$+ ((0.4)^2 x (57 x 12))] = 3426.7 + 2767$
	= 6200 gram
	U
<i>Proposed time</i> $(T) = 28 x 24 x 60 x 60$	<i>Proposed time</i> $(T) = 90 x 24 x 60 x 60$
= 2419200 second	= 7778000 secona
$Mass \ loss = Wi - Wf = Wi \ x \ \rho$	$Mass \ loss = Wi - Wf = Wi \ x \ \rho$
$= 1700 \ x \ 0.12 = 204 \ gram$	$= 6200 \ x \ 0.1 = 620 \ gram$
$I_{corr} = \frac{(Wi - Wf)x F}{\pi x D x L x W x T}$	$I_{corr} = \frac{(Wi - Wf)x F}{\pi x D x L x W x T}$
(204 <i>x</i> 96487)	(620 x 96487)
$I_{corr} = \frac{1}{\pi x 1 x 270 x 27.927 x 2419200}$	$I_{corr} = \frac{1}{\pi x 1 x 1226 x 27.927 x 7776000}$
$= 3.434 x 10^{-4} Amp/cm^2$	$= 7.153 x 10^{-5} Amp/cm^2$
$I_{corr} = \frac{I_{current}}{\pi \ x \ D \ x \ L}$	$I_{corr} = \frac{I_{current}}{\pi \ x \ D \ x \ L}$
$I_{current} = 3.434 x 10^{-4} x 3.1415 x 1 x 270$	I current
= 0.29 Amp	$= 7.153 x 10^{-5} x 3.1415 x 1 x 1226$
	= 0.275 Amp
Use 0.3 Amp for up to 28 days	Use 0.3 Amp for up to 90 days
8.2.3.2 Cleaning of corroded steel coupons and mass loss measurements.

After testing of the RC beams to failure, coupons of corroded steel reinforcement were removed from the tested beams to evaluate the mass loss of steel at the end of corrosion exposure. To remove the corrosion products from the steel there are chemical, mechanical and electrolytic techniques described in the ASTM Standard G1-90. To ensure that the steel bar was free from any adhering corrosion products the rusted steel bars were mechanically cleaned using a stiff metal brush. Stages of removal and cleaning of the corroded steel coupons are shown in Figure 8.15.



Figure 8.15: Stages of removal and cleaning of corroded steel coupons.

To measure the mass loss of the tensile steel reinforcement after exposure of specimens to corrosion, the extracted steel reinforcement coupons were cleaned and measured. The reinforcing bar was then weighed and the percentage mass loss was computed using Eq. (8.5).

$$\text{corrosion mass loss} = \frac{[\text{initial mass} - \text{final mass}]}{\text{initial mass}} x \, 100 \tag{8-5}$$

8.2.4 Mechanical test setup.

All the tested specimens (initial, Repaired and Strengthened RC beams) were loaded under four-point bending with an imposed deflection rate of 0.004 mm/s (with an effective span equal to 1200 mm) using a Zwick testing machine (Figure 8.16). The displacement of the specimens was measured by Linear Variable Displacement Transducers (LVDT) placed at the centre of the specimen for each side. The results include the mode of failure; load carrying capacity and slipping at the interface.



Figure 8.16: Loading setup of the experimental beams (all dimensions in mm).

To record the interface slip at the interface between the strengthening /repair overlaid material and the substrate RC beam during the bending tests, six lateral LVDTs were fixed longitudinal to the interface. The LVDT were attached symmetrically to the beams, three on each side of the load set up (Figure 8.17). Figure 8.18 presents the typical experimental set up of repaired and strengthening beam tests. Each LVDT was glued to the substrate beam and was in contact with a metal angle section that was glued to the strengthened/ repair layer (Figure 8.19). The lateral LVDT were mounted on the concrete surface at the supports and then at incremental distances of 250 mm towards the centre as shown in Figure 8.19. All readings were continuously collected by data-acquisition systems during the test until the failure of beam.



Figure 8.17: Strain gauges layout (all dimensions in mm).



Figure 8.18: Experimental set up showing the distribution of LVDTs for RC beams.



Figure 8.19: Detail of lateral LVDT set up.

8.3 Analysis and discussion of results.

In this section, the experimental results of the beams repaired and strengthened by FRGC overlay are presented and discussed. The results include the mode of failure, load-deflection response and slipping at the interface. Steel mass loss due to corrosion processes are also presented. The results from the corroded beams indicate the effects of corrosion exposure on the flexural response of strengthened and repaired RC beams.

8.3.1 Results of repair techniques.

8.3.1.1 Corrosion damage of control and repaired RC beams.

The visual inspection of the corroded specimens showed that the control specimens had multiple corrosion stains and cracking on the tensile side of the control corroded specimens. During the induced current test, it is assumed that the current applied to the steel bar attracted negatively charged chloride ions from the NaCl solution into the concrete specimens, towards the positively charged reinforcements. As the chloride ions reached the steel-concrete interface above threshold concentrations, the steel surface began to corrode (Sahmaran et al., 2008). The expansive reaction products of the corrosion imposed tensile stresses on the conventional concrete / FRGC cover, resulting in cracking when the tensile stresses exceeded the tensile strength of the cover material. No delamination or spalling of concrete cover was observed. The RC beams repaired with PVAFRGC at different thicknesses (25 mm and 50 mm) showed small and relatively few corrosion stains on the repaired layer as shown in Figure 8.20. Visual inspection confirmed that the majority of the corrosion stains occurred at locations where initial hairline cracks were present, resulting from differential shrinkage. After the flexural test and removal of the overlay repair material, corrosion product stains from rusted steel bars distributed at the interfacial zone of RC beams repaired with 25 mm of PVAFRGC were more common than those for the RC beams repaired with 50 mm of PVAFRGC (Figure 8.21).



Figure 8.20: Tensile side of the control (right) and repaired RC beams (left) after exposure to induced current corrosion.



Figure 8.21: The corrosion of tensile steel bears after specimen demolition.

After demolition of the specimens, the extracted tensile steel reinforcement coupons were cleaned, measured and compared to the non-corroded coupons. The average percentage of steel mass loss was at 8.5%, 7% and 5.5% for control beam and RC beams repaired with 25 mm and 50 mm of PVAFRGC, respectively. These difference in the mass loss of the embedded steel reinforcement could be due to the wide cracks formed in the control specimens, allowing easy migration of corrosion products, seen as rust on the cracked surfaces of the severely damaged specimens (Figure 8.22a). The reduced crack width for RC beams repaired with PVAFRGC prevents movement of the corrosion products, which means that the repair layer acts as a shield against chloride ion migration toward the steel reinforcement, as confirmed by the corrosion products which are seen at the interface area rather than through the repaired layer (Figure 8.22b). Therefore, the PVAFRGC repair layer reduced the corrosion rate of the steel reinforcements remarkably when compared with the conventional concrete cover, at least under the accelerated test conditions used.



Figure 8.22: Side corrosion crack pattern for the corroded control beams (a) and PVAFRGC-corroded beams (b).

8.3.1.2 Mode of Failure and crack pattern of repaired RC beams.

Failure of the control and repaired RC beams at the end of testing are shown in Figures 8.23 to 8.25. Failure of unrepaired beams, both corroded and un-corroded, occurred by flexural crack failure followed by crushing of concrete in the compression zone as shown in Figure 8.23. For the corroded unrepaired specimens, the loss of bonding due to cracking resulting from the volume expansion of the rebar after the electrical acceleration led to spalling of concrete cover at the bottom tensional face of the beam under loading. The RC beams repaired with 25 mm and 50 mm of PVAFRGC overlay failed by rupture of the PVAFRGC layer after yield of the tensile steel reinforcement followed by concrete crushing (Figure 8.24 and Figure 8.25). The accelerated corrosion of the RC beams did not change the failure mode of the repaired material. The same crack pattern was noticed in 25mm and 50 mm layer thickness repaired beams under flexural loading, where multiple cracks are observed in the PVAFRGC overlay layer. The localized large cracks that developed in the substrate RC beam diffused into many fine cracks when they met the PVAFRGC layer, leading to delay in the flexural failure. The cracks start to propagate with increasing loading in a direction perpendicular to the FRGC layer. At the final stage of the loading it was observed that only one crack was completely opened. This is due to the effect of the reinforcing fibres in arresting the cracks, and their stress transfer capability which leads to stress redistribution.



Figure 8.23: Failure of reinforced concrete specimens of control beams.



Figure 8.24: Failure for specimens of repaired beams with 25 mm overlay.



Figure 8.25: Modes of failure for specimens of repaired beams with 50 mm overlay.

8.3.1.3 Loading capacity of repaired RC specimens.

The main loading test results are summarized in Table 8.5. The first crack load for each beam was the load at which the load-deflection relationship for the particular beam was no longer linear and the failure load was determined as 85% of the maximum load. The load versus mid-span deflection curves for each beam series are presented in Figures 8.26-8.28.

	First	crack	Ultima	te point	Failure	Failura	
Specimen ID	Load	Def ^a	Load	Def	Load	Def	railuie
	[kN]	[mm]	[kN]	[mm]	[kN]	[mm]	mode
Ref-1	9.00	0.74	52.50	9.10	44.63	14.50	FL-CC ^b
Ref-2	9.40	0.73	53.40	10.65	45.39	16.00	FL-CC
Ref-corr-1	8.80	0.73	45.20	6.86	38.68	11.00	FL-CC
Ref-corr-2	8.50	0.73	43.30	7.03	37.15	12.13	FL-CC
PVAFRGC-R25-1	12.20	1.13	60.75	8.00	51.64	12.20	FL-CC
PVAFRGC-R25-2	13.80	0.97	61.00	9.00	51.85	16.00	FL-CC
PVAFRGC-R25-corr-1	14.21	1.08	55.00	6.81	46.75		FL-CC
PVAFRGC-R25-corr-2	13.23	1.41	53.54	6.89	45.51	14.25	FL-CC
PVAFRGC-R50-1	15.17	1.37	60.90	7.63	51.77	11.30	FL-CC
PVAFRGC-R50-2	14.80	1.59	60.20	8.33	51.17	12.60	FL-CC
PVAFRGC-R50-corr-1	14.00	1.85	55.47	7.71	46.58	16.80	FL-CC
PVAFRGC-R50-corr-2	14.40	1.71	56.40	8.22	47.94	12.00	FL-CC

Table 8.5: Test results of control (reference) and repaired beams.

^aDef is refer to deflection

^bFL-CC flexural failure followed by secondary compression concrete failure

Reference RC beams.

The load-deflection curves of the control corroded and non-corroded specimens are presented in Figure 8.26 (a-b).



Figure 8.26: Load-deflection curves of reference beams.

The control RC beams had a typical flexural failure followed by secondary concrete crushing (as noted above). From Figure 8.26 and Table 8.5, it can be seen that the first cracking of non-corroded RC beams occurred at a load of 9 kN and 9.4 kN for Ref-1 and Ref-2 specimens, respectively. As the loading increased further, inclined cracks were distributed across the beam with major cracking initiated close to the centre of the beam. The load dropped at 52.5 kN and 53.4 kN and related deflections are 9.1 mm and 10.65 mm for Ref 1 and Ref 2 specimens, respectively. Subjecting the specimens to an induced current of 300 mA for 90 days for steel corrosion with 8.5% steel mass loss had significant effects on the structural response of the control RC

beams (Figure 8.23 c-d). The ultimate load of the corroded RC beams was reduced by 15% and 18.5% for Ref-corr-1 and Ref-corr-2, respectively. The deflection at the ultimate load reduced to 6.86 mm and 7 mm for Ref-cor-1 and Ref-cor-2, respectively (Figure 8.23 b).

PVAFRGC-R25 specimens.

The load-deflection curve of the corroded and non-corroded specimens repaired with 25 mm PVAFRGC overlay are presented in Figure 8.27 (a-b).



Figure 8.27: Load-deflection curves of repaired beam with 25 mm thickness of PVAFRGC material overlay.

As shown in Figure 8.27 and Table 8.5, the failure mode of PVAFRGC-R25 specimens was similar to that of the control RC beams. Application of a 25 mm thickness of PVAFRGC overlay however improved the structural performance of the RC beams. The recorded first cracking load increased by 35% and 50% for PVAFRGC-R25-1 and PVAFRGC-R25-2 specimens compared with the first cracking load of the control beam. As the loading increased further, the cracking distributed across the substrate and the repaired layer increased, and crack width enlarged up to failure. The ultimate failure load increased by 14.6% and 15.5% for PVAFRGC-R25-1 and PVAFRGC-R25-2 respectively compared with control RC beams. The effect of steel corrosion (5.5% steel mass loss) on the structural response of the repaired beams was considerably reduced compared with the control RC beams. For corroded specimens, the ultimate load decreased by 9.5% and 11.5% for PVAFRGC-R25-corr-1 and PVAFRGC-R25-corr-2 respectively compared with the ultimate load value of the non-corroded repaired specimens. Despite corrosion damage and cracking at the interface between the repair overlay and the substrate (Figure 8.24), the ultimate loads of the corroded-repaired specimens were still higher than those of the control noncorroded beam (Ref specimens).

PVAFRGC-R50 specimens.

The load-deflection curve of the corroded and non-corroded specimens repaired with 50 mm PVAFRGC overlay are presented in Figure 8.28 (a-b).





Figure 8.28: Load-deflection curves of repaired beam with 50 mm thickness of PVAFRGC material overlay.

For RC beams repaired with 50 mm thickness of PVAFRGC material overlay, the first cracking load was higher than the respective values of the Ref and PVAFRGC-R25 specimens by 65% and 17% for PVAFRGC-R50-1 and by 61% and 14% for PVAFRGC-R50-2, respectively. However, increasing the repair overlay thickness from 25 mm to 50 mm did not change the ultimate load. With increasing load, multiple cracks in the repair layer were observed, and the crack widths were narrower than that observed in the repaired beam with 25 mm thickness series. The peak load and midspan displacement at failure were 61 kN and 7.65 mm for the PVAFRGC-R50-1 specimen, and were 60 kN and 8.33 mm for PVAFRGC-R50-2 specimen, respectively. The effect of corrosion exposure on the corrosion damage, cracking (Figure 8.25 c-d) and structural response of RC beams (Figure 8.28b) were less pronounced compared with control and repaired beam with 25 mm thickness of PVAFRGC. Visual inspection of the repaired beam after demolition confirmed that no corrosion products were present at the interface between the substrate and the overlay material (Figure 8.21). For corroded RC beams repaired with 50 mm PVAFRGC overlay material, the ultimate failure load decreased to 55.5 kN and 56.5 kN for PVAFRGC-R50-corr-1 and PVAFRGC-R50-corr-2, respectively, which is about a 4%-5% decrease relative to values observed for the PVAFRGC-R50 noncorroded beams (Figure 8.28b).

8.3.1.3.1 Discussion of the repaired RC beams results.

To discuss the structural difference and corrosion resistance between the different thicknesses of the repaired material and control specimens, the comparison of the experimental loads at three points were examined, the first crack loading point, ultimate load and at the failure load of the RC beams. The individual and average of the test results for all RC beams are presented in Figure 8.29 and summarized in Table 8.6 and 8.7.





Figure 8.29: Load against deflection curve comparisons between non-corroded and corroded of initial control beam (a), repaired RC beam with 25 mm of PVAFRGC layer (b) and repaired RC beams with 50mm of PVAFRGC layer (c).

	First crack			Peak load	Failure load		
Non-corroded RC beams	Load	Increase w.r.t. reference beam	Load [kN]	Increase w.r.t. referenc e beam	Def	Load [kN]	Increase w.r.t. reference beam [%]
Ref	9.20		52.95		_ L _ J _	45.01	[```]
PVAFRGC-R25-1	12.20	33	60.75	15	8.00	51.64	15
PVAFRGC-R25-2	13.80	50	61.00	15	9.00	51.85	15
PVAFRGC-R50-1	15.17	65	60.90	15	7.63	51.77	15
PVAFRGC-R50-2	14.80	61	60.20	14	8.33	51.17	14

Table 8.6: Test results of all the non-corroded beams.

Table 8.7: Test results of all the corroded beams.

	First crack			Peak load	Failure load		
Corroded RC beams	Load [kN]	Increase w.r.t. reference beam [%]	Load [kN]	Increase w.r.t. reference beam [%]	Def	Load [kN]	Increase w.r.t. reference beam [%]
Ref-corr	8.65		44.25			37.91	
PVAFRGC-R25-corr-1	14.21	64	55.00	24	6.81	46.75	23
PVAFRGC-R25-corr-2	13.23	53	53.54	21	6.89	45.51	20
PVAFRGC-R50-corr-1	14.00	62	55.47	25	7.71	46.58	23
PVAFRGC-R50-corr-2	14.40	66	56.40	27	8.22	47.94	26

The load details for the non-corroded repaired RC beams are presented in Table 8.6. Compared to the control RC beams, the repaired beams PVAFRGC-R25-1 and PVAFRGC-R25-2 showed first crack load values increased by 33% and 50%, respectively. This means that the stiffness of the repaired RC beams improved by using 25 mm of PVAFRGC in the tension zone. There is also an increase in the ultimate failure load of the repaired RC beams by an average of 15% for both 25 mm and 50 mm repair thicknesses. These results show that the PVAFRGC layer thickness is not a significant factor in improving the ultimate failure load. It can be observed from these results that an additional one-quarter of the initial loading carrying capacity of the RC beam could be achieved by using PVAFRGC with 12.5% of the total beam depth in the tensile side without changing the quantity of longitudinal steel or the cross section of the RC beam.

Applied constant current for 90 days to accelerate the corrosion procedure reduced the ultimate carrying capacity of the control RC beams by 16.5% (Figure 8.29a) and cause 8.5% mass loss of the reinforcements bar. From Table 8.7, increasing the repair layer thickness from 25 mm to 50 mm in the corroded RC beams considerably enhanced the flexural capacity. These results mean that increasing the repair material thickness improved the corrosion resistance which confirmed by the reduction in the mass loss of the corroded reinforcement bar from 7% for PVAFRGC-R25 specimen (Figure 8.29b) to 5.5 % for PVAFRGC-R50 specimen (Figure 8.29c). This behaviour can be explained by two factors: firstly, the superior durability performance of the thicker repair material in terms of corrosion resistance, chloride and moisture penetrations; and secondly, the impact on the interfacial bond between the overlaid repair material and the RC beam substrate, which is the weakest and most critical section of the repaired member. The interfacial bond is influenced by the geometry (thickness) of the repair material, which subsequently effects the corrosion rate of the reinforcement bars and crack damage. A 50 mm thickness of repair layer ensures higher bonding with the concrete substrate by stronger embedding of the longitudinal and shear reinforcement in the repair material. On the other hand, the interfacial bond between 25 mm repair layer and the concrete substrate controlled only by the adhesion through concrete substrate rough surface. This different is also observed by the visual inspection as the stains of the corrosion products distribution at the interfacial area of the PVAFRGC-R25 specimens (Figure 8.21).

8.3.1.4 Interface slip measurement of repaired specimens.

The bond strength at the interface between concrete layers cast at different ages is vital to make sure the monolithic behaviour of the strengthened and repaired reinforced concrete members (Santos, 2009; Júlio *et al.*, 2010). Figures 8.30 and 8.31 illustrate the relationship of load slipping measurements between the RC beam substrate and the overlay repair with 25 mm and 50 mm thickness, respectively. The geometry and the loading of the beams were symmetrical and the preparation of the interface was the same along the whole of its length. A similar set up was followed for all RC beams as mentioned in section 8.2.4 and the slip measurements at the interface between the RC beam substrate and the overlay layer presented here are only due to the bending load, and the effects of creep and shrinkage were ignored.



Figure 8.30 Load vs slipping relationship of repaired RC beams with 25 mm PVAFRGC overlay.



Figure 8.31: Load vs slipping relationship of repaired RC beams with 50 mm PVAFRGC overlay.

From Figure 8.30 and Figure 8.31, the interface slip values are summarized in Table 8.8, which presents data for the maximum load and the respective loads at interface slip values of 0.2 mm $P_{(s=0.2 \text{ mm})}$, 0.8mm $P_{(s=0.8 \text{ mm})}$ and 1.5 mm $P_{(s=1.5 \text{ mm})}$. These are the ultimate accepted slip values for the immediate occupancy, life safety and collapse prohibition behaviour levels respectively according to GRECO (GRECO, 2012; Tsioulou *et al.*, 2013).

Specimen	Р	S_{\max}	$P_{(s=0.2 \text{ mm})}$	$P_{(s=0.8 \text{ mm})}$	$P_{(s=1.5 \text{ mm})}$
	[kN]	[mm]	[kN]	[kN]	[kN]
PVAFRGC-R25-1	60.75	0.75	33.00		
PVAFRGC-R25-2	61.00	1.11	25.00	58.70	
PVAFRGC-R25-corr-1	55.00	0.31	38.00		
PVAFRGC-R25-corr-2	53.54	0.48	40.50		
PVAFRGC-R50-1	60.90	0.73	34.00		
PVAFRGC-R50-2	60.20	0.36	43.00		
PVAFRGC-R50-corr-1	55.47	0.28	25.00		
PVAFRGC-R50-corr-2	56.40	0.98	21.00	50.00	

Table 8.8: Maximum load and slip values for the repaired RC beams.

From the results presented above, it can be observed overall that the slip interface measurements of all beams are small, and increased as cracks began to propagate with an increase in the applied load. For instant, in beam PVAFRGC-R25-1, the interface

slip measurement was very small up to (≤ 0.2) within an applied load of 33 kN, and increased to 0.75 mm at the maximum load of 60.75 kN (Figure 8.30a and Table 8.8). Figure 8.31 illustrates that increasing the repair thickness from 25 mm to 50 mm did not show a significant effect on the interface slip measurements. However, a 50 mm repair thickness delays the respective loads at interface slip values of 0.2 mm, as the first cracking load increased with repair thickness. According to slip measurements for repaired RC beams PVAFRGC-R50-1 and PVAFRGC-R50-2, the respective loads at interface slip values of 0.2 mm were 34 kN and 43 kN, respectively. Exposure of repaired RC beams to accelerated corrosion did not show a major effect on the slip measurement. It can be seen that the interface slip reaches the 1 mm limit only in RC beam PVAFRGC-R25-2 as all specimens failed in by flexural cracking, rather than by separation between the substrate beam and the repaired layer. The interface slip measurement along the beam length for RC beams repaired with 25 mm and 50 mm of PVAFRGC, respectively are presented in Figure 8.32 and Figure 8.33.



Figure 8.32: Interface slip measurements at peak load for repaired RC beams with 25 mm PVAFRGC overlay.



Figure 8.33: Interface slip measurements at peak load for repaired RC beams with 50 mm PVAFRGC overlay.

It can be observed from Figure 8.32 and Figure 8.33 that the interface slip measurements were very low (almost zero) at the supports and the measurements were greater near to the collapse cracks at the mid-span interface slip. Despite the symmetry of the RC beam and the loading conditions, the interface slip measurements were not symmetric as the interface adhesion depends on local conditions, as cracks occur asymmetrically (Tsioulou *et al.*, 2013). Moreover, the debonding of the repaired layer due to differential shrinkage (if there is inadequate connection between the old and the overlay layer) are not included in these results and could affect the slip interface measurement.

Zilch and Reinecke, (2000) stated that load transfer mechanism of shear forces at a concrete to concrete cast in different time interface, according to the shear-friction theory, is composed by: a) adhesion; b) shear-friction; and c) shear reinforcement. According to (ASTM D907, 1996) definition "adhesion is the state in which two surfaces are held together by interfacial forces which may consist of valence forces or interlocking action or both". When the ultimate load is reached, de-bonding take place at the interface and the shear stress will be transferred by mechanical interlocking. If the interface between two concrete layers subject to compression stress, the shear stresses will be transferred by shear-friction. With the relative displacement increment between concrete parts, the reinforcement that crosses the interface will be tensioned

and yielding could happen. Thus, the shear reinforcement will induce compression at the interface and the shear load will be transferred by friction. As slippage occurred, the shear reinforcement will be subjected to shear, named as dowel action (Santos, 2009). There are several analytical models suggested by design codes for the calculation of shear strength at the concrete to concrete interface. The design codes of RC structures to assess the shear strength at the interface presented in this study, are Model Code (2010), Eurocode 2, (2004), ACI Committee 318, (1999), GRECO, (2012) code, and CEB-FIP Model Code, (1990). These design expressions are based on the shear-friction theory, as suggested by Birkeland and Birkeland, (1966), and the following four parameters are considered: a) normal stress at the interface; b) compressive strength of the weakest concrete; c) roughness of the substrate surface; and d) shear reinforcement crossing the interface (Santos, 2009). The interface shear strength of the repaired/ strengthened RC beams can be obtained using the following Eqs. (8.6 - 8.9).

Model code 2010, Eurocode 2 (2004);

$$\tau_{fud} = c.f_{ctd} + \mu.\sigma_N + \rho.f_{yd}.(\mu.\sin\alpha + \cos\alpha) \le 0.5.\nu.f_{cd}$$
(8-6)

ACI Committee 318, (1999) code;

$$\tau_{fud} = \lambda. (1.79 + 0.6. \rho. f_y \le 3.45 MPa$$

$$3.45 MPa \le \tau_{fud} = \pi. \rho. f_{yd} \le \min(0.2. f_{cd}, 5.5 MPa$$
and $f_{yd} \le 414 MPa$
(8-7)

GRECO (2012) code;

$$\tau_{fud} = \begin{cases} 0.25. f_{ct}, & smooth interface \\ 0.75. f_{ct}, & rough interface \\ f_{ct}, & use of shotcrete \end{cases}$$
(8-8)

FIP Model Code 1990;

$$\tau_{fud} = \begin{cases} 0.1 \cdot f_{ct}, & very smooth interface \\ 0.2 \cdot f_{ct}, & smooth interface \\ 0.4 \cdot f_{ct}, & rough interface \end{cases}$$
(8-9)

Where τ_{fud} is the design interface shear strength, f_{ctd} is the design tensile strength of the concrete with the lower strength (between the old and the new concrete) and is

given by $f_{ctd} = f_{ct}/\gamma_c$, where f_{ct} is concrete tensile strength estimated using compressive strength results and a formula proposed in the 2010 Model Code and γ_c = 1.5 for concrete, f_y is steel yield stress, with design value of f_{yd} , σ_N is the external vertical to the interface stress, α is the angle between reinforcement and interface level, c is the adhesion factor, μ is the friction factor (Table 8.9). ρ is the geometric rate of interface reinforcement and λ is a modification factor reflecting the reduced mechanical properties of light- weight concrete relative to normal weight concrete and equals 1 for normal weight concrete.

Table 8.9: value of coefficients c and μ (Eurocode 2 2004).

The respective interface shear stress can be determined according to The British standard BS 8110-1, (1997) using equation (8.10) and the respective results for each repaired beam are presented in Table 8.10.

$$\tau_x = \frac{V_{sd}}{b \cdot z} \tag{8-10}$$

Where τ_x is the interface shear stress of the examined section of the beam according to BS 8110-1 (1997). V_{sd} is the shear force of the examined section of the beam, *b* is the width of the interface, *z* is the lever arm of the composite section.

Table 8.10: The interface shear stress and shear strength of the repaired beams.

Specimen	EC2 and Model Code 2010 [MPa]	ACI-318 [MPa]	GRECO [MPa]	CEB-FIP Model Code 90 [MPa]	τ _x (for Pmax) [MPa]	τ_x (for $P_{(s=0.2 \text{ mm})}$ [MPa]
PVAFRGC-R25-1	0.91	1.79	1.51	0.81	1.69	0.92
PVAFRGC-R25-2	0.91	1.79	1.51	0.81	1.69	0.69
PVAFRGC-R25-corr-1	0.91	1.79	1.51	0.81	1.53	1.06
PVAFRGC-R25-corr-2	0.91	1.79	1.51	0.81	1.49	1.13
PVAFRGC-R50-1	3	3.4	1.51	0.81	1.69	0.94
PVAFRGC-R50-2	3	3.4	1.51	0.81	1.67	1.19
PVAFRGC-R50-corr-1	3	3.4	1.51	0.81	1.54	0.69
PVAFRGC-R50-corr-2	3	3.4	1.51	0.81	1.57	0.58

As can be seen from Table 8.10, the interface shear stress at the respective loads at interface slip values of 0.2 mm in all repaired beams with PVAFRGC layer was lower than the respective interface shear strength in all the examined models apart from FIP Model Code 1990. This can be explained the small value of the slip measurements at this point (s=0.2 mm). While, the maximum shear stress was higher to the respective shear strength model apart from the ACI-318 code in case of PVAFRGC-R25 specimens and ACI-318 code and Eurocode 2 in case of PVAFRGC-R50 specimens. Therefore, the low values of interface slip along the whole length of the interface were due to sufficient adhesion when repaired with PVAFRGC material. For corroded specimens, the shear stress at interface slip values of 0.2 mm and the maximum shear stress was lower than in the non-corroded specimens as the respective bending loads were decreased. The maximum shear stress of PVAFRGC-R50-corr is lower than the shear strength for all examined models apart from FIP Model code 1990. This is also can be explained the small interface slip measurements of the experimental investigations. Moreover, the reaction force at the support points preventing the repaired layer from slipping provides an explanation of the negligible slip measurements at the support point of the beams (Tsioulou et al., 2013).

8.3.1.5 Summary of the repair technique.

In this part of the research, an experimental investigation was undertaken into the effectiveness of a repair technique for the protection of reinforcing steel bars, by replacing the concrete cover with PVAFRGC at different thicknesses (25 mm and 50 mm). Unrepaired specimens were used as control specimens for comparison purposes. The effect of corrosion-induced cracking damage (by applying a nominal 300 μ A constant anodic current for approximately 90 days) on the flexural performance are also evaluated. The results from the corroded and non-corroded specimens are summarized as follows;

• The mass loss of the reinforcement rebar due to corrosion exposure in the control RC beam is about 8.5%. The repaired RC beams showed better corrosion resistance: ca. 7% and 5.5% reduction in mass loss in RC beams repaired with PVAFRGC at thicknesses of 25mm and 50 mm, respectively.

- RC beams with and without a repair layer showed flexural failure modes. Corrosion cracking damage did not change the failure mode of the specimens, i.e. the flexural fracture and the cracks propagate at the mid-span before failure.
- The initial cracking and ultimate load is significantly increased by repairing beams with PVAFRGC. The initial cracking load increased by 43% and 63% for PVAFRGC-R25 and PVAFRGC-R50 specimens, respectively compared to the control RC beam. The ultimate load increased by 15 % for repaired RC beams compared to the control specimens.
- The ultimate load carrying capacity of the corroded control RC beam reduced by 17% compared to the non-corroded control RC beams, while the effect of corrosion on the ultimate load reduced with increasing repair layer thickness. The ultimate load of PVAFRGC-R25-corr and PVAFRGC-R50-corr specimens reduced by 10.5% and 7.4%, respectively compared to the respective non-corroded specimens.
- The interface slip measurement for the corroded and non-corroded specimens showed that in the case of the repair techniques using a new PVAFRGC layer on the tensile side, very small slip measurements were observed along the full length of the beam, which were almost zero at the supports, which means that a strong and effectively bonded interface had developed following roughening and adhesion of the PVAFRGC material to the substrate. Moreover, the slip measurement for the corroded beams indicate that there is a very limited effect of corrosion exposure on the interface slip measurements.

From the summarized points listed above, it is clear that this overlay repair technique has a strong positive effect on the stiffness, the ultimate capacity and corrosion resistance of the RC beams.

8.3.2 Results of the strengthened RC beams.

8.3.2.1 Corrosion damage of strengthened RC beams.

The effect of corrosion exposure on the strengthened RC beams was evaluated through visual inspection of crack distribution and mass loss measurements of the steel reinforcement in the additional strengthening layer. Visual inspection indicated that exposure of RC beams strengthened with conventional techniques generated rust stains and longitudinal corrosion cracking in the side of the strengthening layer parallel to the corroded steel reinforcing bars. RC beams strengthened with SFRGC showed longitudinal cracks at the bottom and side surface of the additional layer, while RC beams strengthened with PVAFRGC did not show any localized cracks (instead multiple tight cracks were observed) in the additional layer. The corresponding crack patterns for the corroded beams strengthened with NSC, SFRGC and PVAFRGC are shown in Figure 8.34.



Figure 8.34: RC beams strengthened with NSC, PVAFRGC and SFRGC after exposure to induced current corrosion and flexural testing.

Figure 8.35 shows the steel reinforcement condition of the repaired beams after corrosion acceleration. The corroded steel reinforcement photos provide examples of the actual corrosion status of reinforcement bars in the additional layer of NSC-S, PVAFRGC-S and SFRGC-S. Corrosion pit formation can be observed on the rebar surface embedded in the NSC layer. In contrast, the reinforcement bars extracted from the additional layers of PVAFRGC and SFRGC had clean surfaces after removal, with less visible corrosion as indicated by a reduction in the weight loss of the rebar.



Figure 8.35: Rebar condition of the strengthened layer the corrosion acceleration.

To quantify differences in the rate of corrosion between the RC beams strengthened with NSC, SFRGC and PVAFRGC, the mass loss measurement is known as the most reliable method to investigate the degree of corrosion (Shaikh and Supit, 2015a). After demolition of the additional strengthened layer, the reinforcement bars were extracted, and then cleaned and measured to determine the mass loss. Average percentages of reinforcement bars mass loss of 11%, 7.5% and 4.5% were recorded after 30 days of accelerated corrosion exposure of RC beams strengthened with NSC, SFRGC and PVAFRGC, respectively. These results indicate that corrosion resistance was increased by using PVAFRGC followed by SFRGC in the retrofitting techniques, rather than normal conventional techniques. The reduction in the corrosion damaged observed for PVAFRGC and SFRGC specimens is due to the decreased chloride penetration depth as indicated in Chapter 7.

8.3.2.2 Mode of Failure and crack patterns of strengthened RC beams.

The failure modes of the strengthened RC beams with additional layers of NSC, PVARGC, and SFRGC are shown in Figures 8.36, 8.37 and 8.38, respectively. The RC beams strengthened by an additional reinforced normal strength concrete layer failed by concrete crushing at the compressive side, followed by shear cracks. For the corroded specimens strengthened by NSC, accelerated corrosion damage and large longitudinal cracks caused spalling of the concrete cover. The corroded beams under

loading failed by shear cracks followed by peeling off of the concrete cover layer i.e. separation of the concrete cover from the additional layer (Figure 8.36 c-d). The RC beams strengthened by the additional PVAFRGC layer failed by crushing concrete at the compression side followed by de-bonding at the interface between the PVAFRGC layer and the substrate concrete (Figure 8.37 a-b). Cracks were widely distributed along the specimens' length with significant bendable performance with increase the applied flexural load, most of these small multiple cracks disappeared and the strengthened beams returned to their initial shape after unloading the specimens. Corrosion exposure of RC beams strengthened by PVAFRGC did not change the failure mode. For the beams strengthened with the SFRGC layer, the failure for the first specimens occurred by shear failure under one of the point loads (Figure 8.38a) and the second beam failed by crushing of concrete at the compression side followed by shearing cracks (Figure 8.38b). The corroded specimens strengthened by SFRGC showed similar cracking patterns under flexural loading. The failure mode of the 3side jacketing strengthened RC beam at the end of test is presented in Figure 8.39. All specimens strengthened by the three-side jacketing technique using PVAFRGC failed in flexural mode, with multiple cracks forming in the PVAFRGC. Then, one of these cracks opened more widely than the others and propagated vertically to the substrate at the centre of the beams.



Figure 8.36: Failure of beams strengthened with a NSC additional layer.



Figure 8.37: Failure of beams strengthened with a PVAFRGC additional layer.



Figure 8.38: Failure of beams strengthened with a SFRGC additional layer.



Figure 8.39. Failure of 3-side jacketing system.

8.3.2.3 Loading capacity of strengthened RC beams.

The main loading test results are summarized in Table 8.11. The first crack load and failure load for each beam was determined as discussed earlier in the repair technique results section (section 8.3.1.3). The load versus mid-span deflection curves for each beam series are presented in Figures 8.40- Figure 8.45.

		First crack		Ultimate point		Failure point		
Strengthening techniques	Specimen ID	Load	Deflection	Load	Deflection	Load	Deflection	Failure mode
		[kN]	[mm]	[kN]	[mm]	[kN]	[mm]	
	NSC-S-1	24.7	1.8	112.28	8.17	95.44	11.68	CC- SH ^a
NSC strengthening	NSC-S-2	22.2	1.7	107.73	7.92	91.57	13.18	CC-SH
RC beam	NSC-S-corr-1	20.5	1.7	66.14	5.79	56.22	10.60	SH-FR ^b
	NSC-S-corr-2	20.0	1.67	71.34	8.22	60.60	10.70	SH-FR
PVAFRGC strengthening RC beam	PVAFRGC-S-1	29.7	3.90	109.00	11.56	92.65	14.45	CC-DB ^c
	PVAFRGC-S-2	30.7	3.13	107.5	11.1	89.03	16.31	CC-DB
	PVAFRGC-S-corr-1	26.9	1.63	110.45	8.14	93.50	9.98	CC-DB
	PVAFRGC-S-corr-2	28.6	1.60	113.55	9.62	96.50	12.70	CC-DB
SFRGC strengthening RC	SFRGC-S-1	35.8	2.02	116.50	8.75	99.03	9.88	Shear
beam	SFRGC-S-2	28.6	1.95	120.80	7.90	102.68	11.30	CC-SH
	SFRGC-S-corr-1	24.3	0.75	118.51	6.79	100.73	13.37	CC-SH
	SFRGC-S-corr-2	21.9	1.19	107.16	7.49	91.08	9.44	CC-SH
PVAFRGC -3SJ	PVAFRGC-3SJ-1	25.0	0.90	76.3	6.3	64.855	15.2	Flexural
	PVAFRGC-3SJ-2	26.6	1.15	73.4	7.16	62.39	12.7	Flexural

Table 8.11. Test results of strengthened RC beams.

o ^aCC-SH refers to concrete crushing at compressive side followed by shear cracks

 $\circ~~^bSH\mbox{-}FR$ refers to shear cracks followed by rupture of the additional layer

o CC-DB refers to concrete crushing at compressive followed by de-bonding

RC beams strengthened with a NSC additional layer.

Figure 8.40 show the load–deflection curves for the RC beams strengthened by using the conventional technique (i.e. using normal strength concrete) for corroded and non-corroded specimens.



Figure 8.40: Load-deflection curves of strengthened RC beam with NSC overlaid.

For non-corroded specimens, the first cracking loads were higher than the control RC beams by 160% and 140% for NSC-1 and NSC-2 specimens, respectively. As loading increased, a diagonal shear crack propagated and the specimens failed by crushing the concrete in the compression zone. After reaching the maximum load 112.3 kN and

107.73 kN for NSC-1 and NSC-2 specimens respectively, the load decreased and the failure loads were approximately 95.5 kN and 91.6 kN, which are around 85% of the ultimate load. As shown in Figure 8.40b, the first and ultimate loads in the corroded specimens decreased sharply, which can be ascribed to damage and cracking of concrete at the additional layer caused by corrosion, which reduced the bonding of the reinforcement bars in concrete and thus compromised the effectiveness of the NSC strengthening system. The ultimate load decreased by 40% and 35% for NSC-S-corr-1 and NSC-S-corr-2, respectively, compared with the non-corroded NSC strengthening RC beams. At high levels of corrosion, the failure under loading started at the longitudinal cracks in the additional layer followed by diagonal shear cracks in the substrate beam (Figure 8.41).



Figure 8.41: Crack patterns of the RC beam strengthened with NSC.

RC beams strengthened with an additional PVAFRGC layer.

The load-deflection curve of the corroded and non-corroded specimens strengthened with 50 mm PVAFRGC overlay are presented in Figure 8.42 (a-b), respectively.


Figure 8.42: Load-deflection curves of strengthened beam with 50 mm thickness of PVAFRGC material overlay.

The RC beams strengthened with an additional layer of PVAFRGC show a significant increase in the flexural capacity. From Figure 8.42a and Table 8.11, it can be seen that the flexural strengthening of the non-corroded specimens with a PVAFRGC additional layer increased the first cracking load by 3 times compared with the control RC beams. The ultimate load increased from 53 kN for the control RC beam to 109 kN and 107.5 kN for PVAFRGC-S-1 and PVAFRGC-S-2, respectively. Flexural failure in the RC

beam strengthened with PVAFRGC occurred at the interfacial bond with the concrete substrate. The PVAFRGC overlay showed multiple cracking behaviour with small crack spacing, and most of these cracks disappeared after unloading the specimens. Figure 8.42b indicates that even following corrosion damage and cracking, the flexural capacity of the specimens strengthened with PVAFRGC was higher than that of the non-corroded NSC strengthened specimens. The first cracking loads were approximately 26.85 kN and 28.6 kN for PVAFRGC-S-corr-1 and PVAFRGC-S-corr-2 specimens, respectively. Diagonal shear cracks start to develop with loading in the substrate concrete beam. As the load increases, the critical flexural-shear crack propagated. Subsequently, crack opening takes place between the interfacial zone of the PVAFRGC layer and the existing concrete, and crushing of the upper edge concrete at loads of 110.5 kN and 113.6 kN for PVAFRGC-S-corr-1 and PVAFRGC-S-corr-2 specimens, respectively (Figure 8.43).



Figure 8.43: Crack pattern distribution in the strengthened RC beam.

After corrosion exposure, very small multiple cracking rather than localized larger cracks was observed in the PVAFRGC overlay generated by corrosion expansion. These tight cracks slow down further chloride ion penetration and reduce the rate of corrosion propagation. This behaviour is in agreement with the material durability results presented in Chapter 7. The effective higher resistance of the PVAFRGC material to the access of chloride ions and moisture to the reinforcing bar is a significant benefit of PVAFRGC over normal strength concrete and mortar.

RC beams strengthened with an additional SFRGC layer.

The load-deflection curves of corroded and non-corroded RC beams strengthened with SFRGC additional layer are shown in Figure 8.44.



Figure 8.44: Load-deflection curves of strengthened beam with 50 mm thickness of SFRGC material overlay.

From Figure 8.44a and Table 8.11, the non-corroded RC beams strengthened with an SFRGC layer showed an increase in the first cracking load by 290% and 321% for

SFRGC-S-1 and SFRGC-S-2, respectively, relative to that of the control RC beam. As the loading increased, a diagonal shear crack propagated under one of the point loads (Figure 8.38a) while the second specimen failed by crushing concrete in the compression zone followed by diagonal shear cracks. The ultimate failure load values were 116.5 kN and 120.8 kN for SFRGC-S-1 and SFRGC-S-2, respectively. Results from SFRGC-S-corr specimens (Figure 8.44b) indicate that the 4.5% reduction in the mass of the steel reinforcement in the additional layer slightly reduced the beam's flexural capacity. The reduction in flexural capacity of the corroded strengthened RC beams was approximately 34% and 38% for SFRGC-S-corr-1 and SFRGC-S-corr-2, respectively, at the first cracking, while no reduction was observed in the ultimate load of the SFRGC-S-corr-1 specimen, and only a minor (around 6%) reduction was recorded in the SFRGC-S-corr-2 specimen.

Strengthened beams with 3 side jacketing with PVAFRGC

Figure 8.45 show the load-deflection curves for RC beam strengthened with 3 side PVAFRGC jacketing. Strengthening U-jackets were applied to two RC beams (Figure 8.46).



Figure 8.45: Load-deflection curves of RC beams strengthened with PVAFRGC jacketing.

The RC beams strengthened with PVAFRGC jacketing have considerably improved load carrying capacities compared to the control beam. The improvement in the load values for PVAFRGC-3SJ-1 and PVAFRGC-3SJ-2 were 170% and 195% at the first crack load point, while increase in the peak load was 44% and 38.4% compared to the control RC beams. The load deflection curve shows that three stages characterize the structural response: an elastic stage, a second stage where stiffness is governed by multiple cracking in the jacketing layer, and a third softening branch, whose slope is due to macro-crack localization. After reaching the ultimate load value of 75 kN, the load decreased and stabilized with a horizontal branch at, approximately, 62-66 kN with localization of a single flexural crack and remain higher than the ultimate load of the control RC beam. Failure occurred due to yielding of the longitudinal reinforcement, with the final flexural crack pattern shown in Figure 8.39.



Figure 8.46: PVAFRGC-3SJ beam under flexural test (left) and beam cross section (right).

8.3.2.3.1 Discussion of the strengthened RC beam results.

The strengthened RC beam results were compared with the control beams and the RC beams strengthened with NSC to evaluate the structural performance of the PVAFRGC and SFRGC strengthening system. Loads at two points (at the first crack loads and at the peak loads) are considered for discussion purposes. A comparison of the load deflection curve of the non-corroded and corroded strengthened beams is presented in Figure 8.47 and the non-corroded RC beam strengthened with 3 side jacketing is presented in Figure 8.47 and summarized in Tables 8.12 and 8.13.





Figure 8.47: Load against deflection curve comparisons between corroded and noncorroded strengthened RC beams with NSC layer (a), SFRGC layer (b) and PVAFRGC layer (c).



Figure 8.48: Load against deflection curve comparisons between the initial RC beams and RC beams strengthened with 3 sides jacketing.

Specimen ID		First crac	k load	Peak load			
	Load [kN]	Increase w.r.t. reference beam [%]	Increase w.r.t. NSC-S specimens [%]	Load [kN]	Increase w.r.t. reference beam [%]	Increase w.r.t. NSC-S specimens [%]	
Ref	9.20			52.95			
NSC-S	23.46	155		109.64	107		
PVAFRGC-S-1	29.68	223	27	109.00	106	-1	
PVAFRGC-S-2	30.65	233	31	104.72	98	-4	
SFRGC-S-1	35.81	289	53	116.50	120	6	
SFRGC-S-2	38.72	321	65	120.80	128	10	
PVAFRGC-3SJ-1	25.00	172	7	76.30	44	-30	
PVAFRGC-3SJ-2	26.61	189	13	73.40	39	-33	

Table 8.12. Test results of the non-corroded strengthened RC beams at first crack load and at peak load.

Table 8.13. Test results of the corroded strengthened RC beams at first crack load and at peak load.

		First crack lo	ad	Peak load			
Specimen ID	Load	Increase w.r.t. reference beam	Increase w.r.t. NSC specimens		Increase w.r.t. reference beam	Increase w.r.t. NSC specimens	
	ןאוז	[/0]	[/0]		[/0]	[/0]	
Ref-corr	8.6			44.3			
NSC-S-corr	20.2	134		68.7	55		
PVAFRGC-S-corr-1	26.8	210	33	110.5	150	61	
PVAFRGC-S-corr-2	28.6	231	41	113.5	157	65	
SFRGC-S-corr-1	24.5	183	21	118.5	168	72	
SFRGC-S-corr-2	22.8	163	13	111.4	152	62	

At the first crack point, compared to the reference beams, the strengthened beams with NSC, PVAFRGC and SFRGC increase the load value by ca. 155%, 228% and 305%. The stiffness of the RC beams strengthened with NSC and SFRGC layers is higher than in PVAFRGC-S strengthened specimens (Figure 8.47). The average peak load values increased by 107%, 102% and 124% for NSC-S, PVAFRGC-S and SFRGC-S specimens compared to the control beams (Table 8.12). The behaviour of the beams strengthened with the reinforced additional layer were very similar between different

materials, with optimum structural performance observed in beams strengthened with SFRGC. This is due to the presence of $2\Phi 10$ mm reinforcement bar in all the additional layer which attributed to carry the most of flexural capacity of RC beams. However, the deflection at the peak load of PVAFRGC-S specimens was around 11.2 mm higher than NSC-S and SFRGC specimens 8.32 mm and 8.05 mm, respectively.

For the corrosion damaged specimens, the average of the first crack load of the RC beam strengthened with NSC considerably reduced by approximately 16%, while the average peak load value reduced by 37.5% relative to the non-corroded specimens (Figure 8.47a). In contrast, despite corrosion damage, the PVAFRGC-S-corr strengthening system increased the average peak load values by 5% compared with the non-corroded PVAFRGC-S specimens. The ultimate failure load values of corroded and non-corroded RC beams strengthened with SFRCG layers were quite similar, at 118.7 kN and 115 kN, respectively (Figure 8.47c). Compared to the NSC-S-corr, the first crack load values increased by 37% and 17% for PVAFRGC-S-corr and SFRGC-S-corr specimens, respectively, on average. The peaks load of the PVAFRGC-S-corr and SFRGC-S-corr were higher than the NSC-S-corr specimens by 63% and 67.3%, respectively. The strengthened RC beams with non-reinforced PVAFRGC jacketing improved the load values by 172% and 189% at first crack load, while the increase in the peak load was 44% and 39%, respectively compared to the reference RC beam (Figure 8.48).

A comparison between RC beams strengthened with NSC, PVAFRGC and SFRGC specimens revealed that increasing the compressive strength of the strengthening layer from 43 MPa to 70 MPa had little influence on the flexural capacity of the strengthened RC beams, however, RC beams strengthened with SFRGC and PVAFRGC significantly enhanced the corrosion resistance of the RC beams. Accumulated corrosion remnant around the reinforcement bar increased the pressure on the surrounding concrete in all directions. At the early stage of corrosion, internal pressure generally strengthened the bond between reinforcing bar and concrete. These positive effects of slight corrosion observed on the load-deflection curves of the strengthened RC beams with SFRGC and PVAFRGC after 7.5% and 4.5% mass loss, respectively. This enhancement can be attributed to the fact that when the FRGC overlay is used, the crack widths generated by the corrosion products surrounding the reinforcement

bars are reduced. Cracking, particularly large cracks, allow the conductive chloride solution to come into direct contact with the steel surface, consequently providing a direct current path between the steel reinforcement bars and the electrodes and resulting a reduction in electrical resistance following cracking in the cementitious material around the steel bar (Sahmaran *et al.*, 2008; Sahmaran *et al.*, 2015).

8.3.2.4 Interface slip measurement of strengthened specimens.

Figures 8.49, 8.50 and 8.51 illustrate the relationship between load and slip measurement in the RC beams strengthened with NSC, PVAFRGC and SFRGC, respectively. The geometry of the strengthened overlay was symmetrical and the preparation of the interface was the same along the whole of its length. Thus, the slip measurements against the load are controlled by the differential crack development at the interface with different overlay materials under flexural loading.



Figure 8.49: Load versus slipping relationship of RC strengthened with NSC overlay.



Figure 8.50: Load versus slipping relationship of RC strengthened with PVAFRGC overlay.



Figure 8.51: Load versus slipping relationship of RC strengthened with SFRGC overlay.

From Figure 8.49, Figure 8.50 and Figure 8.51, the interface slip values are summarized in Table 8.14, which shows the maximum load (P) and the respective

loads at interface slip values of 0.2 mm $P_{(s=0.2 \text{ mm})}$, 0.8mm $P_{(s=0.8 \text{ mm})}$ and 1.5 mm $P_{(s=1.5 \text{ mm})}$ as mentioned earlier in section (8.3.1.3) (GRECO, 2012; Tsioulou *et al.*, 2013).

Specimen	Р	S _{max}	P(s=0.2 mm)	P(s=0.8 mm)	P _(s=1.5mm)
	[kN]	[mm]	[kN]	[kN]	[kN]
NSC-S-1	106.82	1	60	87	
NSC-S-2	112.16	0.69	60.7		
NSC-S-corr-1	66.14	0.32	45.68		
NSC-S-corr-2	71.34	1.33	36.75	65.7	
PVAFRGC-S-1	109	0.7	57.9	109	
PVAFRGC-S-2	104.72	0.41	67.4		
PVAFRGC-S-corr-1	110.45	0.82	80.1	80.6	
PVAFRGC-S-corr-2	113.55	0.6	79.31		
SFRGC-S-1	116.5	0.462	87.83		
SFRGC-S-2	120.8	0.336	78.24		
SFRGC-S-corr-1	118.51	0.26	82		
SFRGC-S-corr-2	111.44	0.6	50		

Table 8.14. Maximum load and slip values for the strengthened RC beams.

According to the slip measurements of the RC beams strengthened with NSC overlay (Figure 8.49 and Table 8.14), the maximum interface slip measurements varied depending on the failure mode and crack propagation along the RC beams substrate and the overlay material. For example, the slip measurements at the ultimate load for NSC-1 and NSC-2 specimens was 2.13 mm and 0.69 mm and the respective load at interface slip values of 0.2 mm were 60 kN and 60.7 kN, respectively. The slip measurements of the corroded specimens showed similar behaviour to the non-corroded specimens (Figure 8.49 c and d). However, the respective loads to achieve 0.2 mm were smaller than in non-corroded specimens (Table 8.14). This is due to the initial cracks along the reinforcement of the strengthened layer (resulting from corrosion exposure) generating crack propagation in the middle of the strengthened layer, rather than separation between the initial beam and the strengthening layer. The interface slip measurements reach the 1.2 mm limit only in the NSC-S-corr-2 specimen (Figure 8.49 d).

Figure 8.50 illustrates the relationship between load and slip measurements of the noncorroded and corroded RC beams strengthened with PVAFRGC overlay. In all RC beams strengthened with PVAFRGC interfacial slip measurements are less than 0.8 mm, apart from the PVAFRGC-S-corr-1 specimens (Figure 8.50c) as the slip suddenly increased from 0.38 mm to 0.82 mm close to the failure load of 109 kN. This is due to crack opening which takes place at the interfacial zone between the PVAFRGC layer and the existing concrete at the ultimate failure load.

Figure 8.51 and Table 8.14 show the slip measurements of RC beams strengthened with SFRGC. Similar trends were observed to the RC beams strengthened with NSC and PVAFRGC overlay. For SFRGC-S-1 and SFRGC-S-2 specimens, the slip measurements at the ultimate failure load were 0.46 mm and 0.34 mm, and the respective loads at 0.2 mm were 87.83 kN and 78.24 kN, respectively. Exposure of specimens to accelerated corrosion did not significantly affect interface slip measurements. For corroded specimens SFRGC-S-corr-1 and SFRGC-S-corr-2, the maximum slip measurements were 0.26 mm and 0.6 mm, and the respective loads at 0.2 mm were 87.94 kN and 50 kN, respectively. The interface slip measurement along the beam length for RC beams strengthened with NSC, PVAFRGC and SFRGC respectively are presented in Figures 8.52, 8.53 and 8.54.



Figure 8.52: Interface slip measurements at peak load for strengthened RC beams with NSC overlay.



Figure 8.53: Interface slip measurements at peak load for strengthened RC beams with PVAFRGC overlay.



Figure 8.54: Interface slip measurements at peak load for strengthened RC beams with SFRGC overlay.

By comparing Figures 8.52, 8.53 and 8.54, it can be summarized the experimental results that the slip measurements for all beams were very small along the whole length of the of the interface, and within acceptable limits compared with the GRECO standard (GRECO, 2012). The slip measurements at the supports were found to be the smallest interface value, and increased as bending cracks began to propagate through the interface towards the mid-span of the beam. Moreover, there is somewhat scatter on the interface slip measurements in the duplicate specimens and did not behave

monolithically along the whole length of the specimen due to the slip measurement depending on the cracks propagation and failure mode.

Table 8.15 presents the maximum values of the interfacial shear strength and shear stress of the strengthening RC beams. All data needed to calculate the interface shear strength using Eqs. (8.6-8.9) and the the interface shear stress using Eq (8.10) was determined for the maximum load and the respective load at the interface slip and the respective loads at interface slip values of 0.2 mm $P_{(s=0.2 \text{ mm})}$.

	EC2 and	ACI-	GRECO	CEB-	τ_x (for	τ_x (for
	Model	318		FIP	P _{max})	$P_{(s=0.2 mm)})$
Specimen	Code			Model		
	2010			Code 90		
	[MPa]	[MPa]	[MPa]	[MPa]	[MPa]	[MPa]
NSC-S-1	0.91	1.79	1.51	0.81	2.67	1.50
NSC-S-2	0.91	1.79	1.51	0.81	2.80	1.52
NSC-S-corr-1	0.91	1.79	1.51	0.81	1.65	1.14
NSC-S-corr-2	0.91	1.79	1.51	0.81	1.78	0.92
PVAFRGC-S-1	0.91	1.79	1.51	0.81	2.73	1.45
PVAFRGC-S-2	0.91	1.79	1.51	0.81	2.62	1.69
PVAFRGC-S-corr-1	0.91	1.79	1.51	0.81	2.76	2.00
PVAFRGC-S-corr-2	0.91	1.79	1.51	0.81	2.84	1.98
SFRGC-S-1	0.91	1.79	1.51	0.81	2.91	2.20
SFRGC-S-2	0.91	1.79	1.51	0.81	3.02	1.96
SFRGC-S-corr-1	0.91	1.79	1.51	0.81	2.96	2.05
SFRGC-S-corr-2	0.91	1.79	1.51	0.81	2.79	1.30

Table 8.15. The interface shear stress and shear strength of the strengthened RC beams.

Comparing the shear stress with shear strength in the strengthening beam with NSC overlay, it can be seen that interfacial shear stress of non-corroded specimens is higher than the shear strength of Model code 1990 and Eurocode 2 at the respective load at interface slip values of 0.2 mm $P_{(s=0.2 \text{ mm})}$. While, the maximum shear stress was higher than shear strength measured according to all the examined design codes. For the strengthening RC beams with PVAFRGC and SFRGC layer, the shear stress of corroded and non-corroded specimens at the respective load at interface slip values of 0.2 mm $P_{(s=0.2 \text{ mm})}$ and maximum load were higher than shear strength of all the examined model codes. Thus, the high values of interface slip between the supports, which are much higher than the limit of 0.02 mm as the shear resistance due to interface adhesion significantly lower than the shear stress. While the small slip

measurements at the supports can be explained due to interface shear resistance together with any additional friction at the supports due to a normal to the interface stress.

8.3.2.5 Summary of the strengthening technique.

In this section of the Chapter, an experimental investigation was carried out into the performance of RC beams strengthened using a thin reinforced layer of NSC, PVAFRGC and SFRGC. The application of a PVAFRGC jacket on a RC beam was also investigated. The effect of severe environmental conditions on the flexural capacity of RC beams was evaluated by exposing RC beams to accelerated induced current. The results obtained in this section of the study can be summarized as follows:

- A PVAFRGC and SFRGC strengthening layer considerably reduced the effect of corrosion exposure on the mass loss of the reinforcement bar, cracks distribution and flexural performance compared to the control RC beams and RC beams strengthened with NSC. Corroded NSC-S specimens showed a 37% reduction in load carrying capacity, while the corroded SFRGC specimens showed only a 3% reduction. There was no significant reduction in flexural performance of the corroded PVAFRGC specimens.
- The application of PVAFRGC for 3 side jacketing on a RC beam generates an increase in load bearing capacity. The ultimate load increased by 50% compared to the control RC beams.
- The application of a reinforced additional layer of NSC, PVAFRGC and SFRGC for the strengthening of RC beams is very efficient, as the ultimate loading increase is about 2 times compared to control RC beams.
- Interface slip measurements were very small for all specimens, and the effect of corrosion on the interface slip was negligible.
- The strengthening technique using PVAFRGC and SFRGC materials provides a significant structural enhancement at the serviceability limit state resulting from a remarkable increase in the beam service load and stiffness, and increased durability, due to reduced crack widths and low permeability of the FRGC material.

• The technology of FRGC application is relatively simple (and novel): curing at ambient temperature and humidity is sufficient to allow the development of the strength characteristics of the FRGC, the material can be cast in a thin layer due to high fluidity, and a simple roughening of the beam surface ensures good adhesion of the layer and jacket without using any adhesive material.

CHAPTER 9:

CONCLUSION AND RECOMMENDATIONS

9.1 Summary.

This investigation has provided new insights into the development of high strength fibre reinforced geopolymer concrete cured under room temperature. Experimental investigations were carried out which resulted in quantitative data on the mechanical and durability properties of plain and fibre reinforced geopolymer composites. The study consisted of four main phases. The first phase of the study involved characterising the fresh and hardened behaviour of binary and ternary blended geopolymer mortar. Different compositions of fly ash, slag and different silica fume particle size were utilized in this study. The second phase included an extensive experimental evaluation of the effectiveness of randomly distributed fibre types and volume fractions in improving the load carrying capacity and strain hardening properties of ambient temperature cured geopolymer materials. Five types of fibres: 6 mm straight steel, 13 mm straight steel, hooked end steel, PVA, and glass fibres were investigated at different volume fractions. The third phase studied the influence of randomly distributed fibre types on the durability performance of fibre reinforced geopolymer concrete. Four experiments were conducted, shrinkage (free shrinkage, end restrained shrinkage and overlay restrained shrinkage), corrosion resistance, rapid chloride migration test and chemical resistance (sulphuric acid and sodium sulphate). The fourth phase focused on evaluating the structural performance of FRGC for strengthening and repair of existing reinforced concrete beam elements. The following conclusions can be drawn based on the results obtained from the experimental investigations.

9.2 Overall Conclusions.

9.2.1 Fresh properties of plain geopolymer.

The mixing procedure significantly affected the fresh properties of the geopolymer material. Mixing the solid components together first followed by adding the liquid solution to the solid binder enhanced the performance of the final product. The setting time and workability increased as the water content and polycarboxylate- based superplasticizer percentage were increased.

Overall it was seen from the binary and ternary blended geopolymer mortar that water demand generally increased with the addition of GGBS to the mixture. Increasing the slag content in the fly ash and slag based geopolymer mortar decreases the workability and accelerates the setting times (initial and final) and mortar hardening. The inclusion of silica fume in the geopolymer mortar has various effects on the flow characteristics of fly ash and slag based geopolymer mortar. When replacing fly ash with undensified and slurry silica fume, the workability and setting time were considerably reduced compared to the control fly ash and slag mixture. On the other hand, the larger particle size of silica in densified silica fume did not significantly affect workability.

9.2.2 Mechanical and microstructural properties of plain geopolymer mortar.

Hardened fly ash and slag based geopolymer mortar with high strength properties can be produced without elevated heat curing. Curing conditions (temperature and time) have clear effects on the behaviour of geopolymer mortar. The 28-day compressive strength of the specimens cured at room (ambient) temperature is close to the strength of the respective specimens cured under heat treatment. However, elevated temperature significantly improved the early age strength compared with the respective mixture cured under ambient temperature. The optimum strength of fly ash and slag based geopolymer mortar was obtained when the alkaline solution, water and superplasticizer weight to total binder ratios were 12%, 25% and 1%, respectively.

Compressive, flexural and tensile strength of geopolymer mortar cured under room temperature considerably improved by increasing slag content from 10% to 40% of the total binder. Inclusion of undensified and slurry silica fume improved the compressive strength of plain geopolymer mortar, while the compressive strength slightly decreased with the addition of densified silica fume.

Microstructural observation by scanning electronic microscopy confirms that the incorporation of slag and silica fume as a partial fly ash replacement in geopolymer mortars densified the microstructure, leading to an improvement in mechanical strength. The images show a less dense structure and non-reacted fly ash particles with low contents of slag. However, the compactness of the geopolymer matrix increased

when slag content was increased. FTIR analysis allowed assessment of the degree of geopolymerization and the formation of reaction products. The addition of slag affected the structural reorganisation by increasing C-S-H gel formation and reducing the amount of aluminosilicate gel related to fly ash, which in turn accelerates geopolymerisation. Thermal analysis (DSC) showed that the addition of slag increased the heat flow in the samples and reduced the time to achieve the main thermal (reaction) peak due to accelerated formation of geopolymerization gels, leading to a reduction in the setting time.

9.2.3 Mechanical and microstructural properties of fibre reinforced geopolymer concrete.

The experimental investigations showed that geopolymer matrix compositions considerably affected the mechanical properties of steel fibre reinforced geopolymer concrete cured under room temperature. The compressive strength and tensile strength performance of SFRGC was considerably enhanced by increasing the mechanical strength of the geopolymer matrix. The Young's modulus, ultimate tensile strength and energy absorption capacity significantly improved with increasing slag content and inclusion of fine particle sizes of silica fume. At lower slag to binder weight ratio, replacement of fly ash with undensified silica fume increased the compressive and tensile strength of SFRGC. However, using the DSF form in the SFRGC composite slightly reduced the compressive strength. The results also indicated that as the age of geopolymer specimens increased from 3 days to 90 days, compressive strength and tensile strength were significantly improved for all the examined mixtures. For geopolymer specimens cured under room temperature, the geopolymerization process considerably improves with sufficient curing time, leading to improvements in the strength of the geopolymer matrix and enhancements of the bond between the geopolymer matrix and the reinforcement fibre.

The addition of discontinuous fibre to geopolymer mortar had differing effects depending on fibre type, aspect ratio and volume fraction. Inclusion of steel fibre improved the mechanical properties of the examined mixtures in terms of compressive strength, tensile strength and post crack behaviour. The results showed that increasing straight steel fibre length from 6 mm to 13 mm and volume fraction from 1% to 3%

significantly developed the deflection hardening behaviour. The deflection hardening behaviour of a SFRGC specimen with 3% dosage of steel fibre with 13 mm length gave almost twenty times higher deflection at the peak load than specimens without fibre and 4 times that of 3% of steel fibre with 6 mm length. The experimental results also showed that hybrid macro hooked end steel fibres with micro steel fibre resulted in improvement of mechanical strength and deflection compared to single macro steel fibre type additives. The ultimate flexural strength of hybrid 1% V_f HE fibre with micro steel fibre (1% and 2% V_f) are 2 and 4 times larger than the ultimate strength of macro 1%HE alone.

Addition of PVA fibres to the geopolymer mortar did not give the same pronounced improvement in the compressive strength. However, the tensile and flexural strength performance was considerably improved compared to the plain geopolymer mortar. The mechanical properties of PVA fibre reinforced geopolymer composite significantly developed with increasing dosage rate from 1% to 2%. The ultimate tensile and flexural strength increased from 2.7 MPa and 8.5 MPa at 1% PVAFRGC to 3.5 MPa and 8.9 MPa, respectively. The deflection at the ultimate flexural load considerably increased from 2.2 mm to 5.5 mm with increased fibre dosage rate (from 1% to 2% V_f)

The flexural toughness of different fibre reinforced geopolymer concrete mixtures was evaluated by combining ASTM C1018 and ASTM C1609 standard codes at six deflection points ($3\delta_1$, $5.5\delta_1$, $10.5\delta_1$, L/600, L/150, and L/100) beside the first and second peak loads. The toughness at all deflection points increased with increasing aspect ratio and volume fraction of steel fibre. A 3% volume fraction of 13 mm steel fibre mixture showed the highest toughness values of around 4.4 joule, 28 joule, 66 joule, and 120 joule at δ_1 , $3\delta_1$, $5.5\delta_1$, $10.5\delta_1$, respectively. Toughness of the hybrid 1 % V_f of HE fibre with micro fibre (1% and 2% V_f) is 3-14 times higher for the first cracking and 3.6-5.3 times higher at second cracking than for specimens containing hooked end fibres alone.

Toughness, and toughness indices at the peak load, of PVAFRGC significantly increased by increasing the fibre volume fraction. 2% V_f of PVAFRGC exhibited highest toughness index values of 84 at I_{peak} proving the superior deflection hardening

behaviour of this composite material. The strong bond between PVA and steel fibre and the geopolymer matrix is the major toughening mechanism in the geopolymer composite as indicated by the microstructural analysis (SEM section, below) in this study. Thus, it can be concluded that all ST13, HE and PVA fibre reinforced mixtures exhibited considerably improved post-cracking flexural performance, which is even superior to perfectly plastic behaviour. At the second peak load, PVAFRGC exhibited the highest energy absorption capacity. The order of performance of different fibre types at this deflection level was as follows: PVA-fibres > ST13-fibers > glass-fibres.

9.2.4 Durability properties of plain and fibre reinforced geopolymer concrete.

The durability performance of plain geopolymer and fibre reinforced geopolymer composite cured under room temperature have been investigated and compared with the conventional Portland cement mortar. The durability characteristics of fibre reinforced geopolymer containing 3% ST13, 2% PVA, 1% Glass fibres have been examined in term of shrinkage characteristics, corrosion resistance, rapid chloride migration test, and chemical resistance.

The shrinkage performance of plain and fibre reinforced geopolymer concrete cured under room temperature have been evaluated through drying shrinkage, overlay restrained shrinkage and end restrained shrinkage. Shrinkage in the plain geopolymer mixture was much higher than in ordinary Portland cement mortar, whereas inclusion of steel, PVA and glass fibre in the geopolymer mixture significantly reduced the drying shrinkage by 45%, 53% and 70%, respectively. Moreover, overlay and end restrained shrinkage indicated that steel and glass fibre reinforced geopolymer composite had a significant effect in controlling cracking in terms of crack width, and time before cracking, compared to the plain of geopolymer mortar. Restrained shrinkage of the PVAFRGC overlay material was higher than SFRGC and GFRGC overlay with multiple cracking behaviour.

An accelerated corrosion testing technique was employment to accelerate the corrosion and shorten the test period. The corrosion process was initiated by applying a constant current of 300 mA to steel embedded in concrete prisms after 6 days and 12 days curing time. Visual inspection showed that induced corrosion caused large

cracks along the length of the conventional mortar specimen, and the crack width increased with time as corrosion activity progressed. On the other hand, the cracks in SFRGC and PVAFRGC specimens were very small and remained almost constant with time as corrosion activity progressed. Flexural testing of the prisms after exposure to corrosion indicated that load carrying capacity was reduced in conventional mortar prisms, while corrosion exposure of FRGC prisms had only a very small effect on the flexural load carrying capacity.

A rapid chloride migration test was conducted according to non-steady state chloride migration (NT Build 492) to investigate the resistance of conventional mortar, plain geopolymer mortar and FRGC specimens against chloride penetration. The results indicated that plain geopolymer mortar and FRGC specimens cured under room temperature have higher resistance against chloride penetration than conventional mortar. The chemical resistance of plain geopolymer mortar and FRGC specimens has been evaluated by immersing cubic specimens in 3% of sodium sulphate and sulphuric acid for up to 6 months. The results showed that plain geopolymer and FRGC specimens have an excellent resistance to sodium sulphate and sulphuric acid in term of compressive strength reduction and mass loss compared to the conventional mortar cube specimens.

9.2.5 Application of FRGC for repair and strengthening RC beams.

The structural performance of SFRGC and PVAFRGC has been evaluated by experimental investigation of repaired and strengthened reinforced concrete beams. Twenty-six RC beams were constructed using Portland cement concrete with compressive strength equal to 32 MPa. The technique of applying FRGC overlay on the RC beams is relatively simple in terms of surface preparation by roughening of the beam surface ensures good adhesion of the layer and jacket without using any adhesive material, curing at ambient temperature and humidity is sufficient to allow the development of the strength characteristics of the FRGC. The effect of corrosion-induced cracking damage on flexural performance was assessed following applying a nominal 300 μ A constant anodic current. The results from the corroded and non-corroded specimens are summarized as follows;

9.2.5.1 Repair of RC beams by PVAFRGC.

The effectiveness of a PVAFRGC repaired layer as a barrier against chloride ion penetration to steel reinforcement bar has been evaluated by replacing the concrete cover on the tensile side with PVAFRGC. Two different repair thicknesses of 25 mm and 50 mm have been evaluated. The test results showed that the mass loss of the steel reinforcement from corrosion exposure was significantly reduced in the RC beam repaired with PVAFRGC. All the corroded and non-corroded RC beams failed in flexural testing and the cracks propagated at the mid-span before failure. The flexural load carrying capacity of the repaired RC beam considerably increased by 15% compared to the control non-repaired RC beam. Increasing the PVAFRGC repair thickness from 25 mm to 50 mm did not exhibited any significant increase in the load carrying capacity of the repaired RC beams. However, the effect of corrosion exposure on the ultimate load carrying capacity was considerably reduced by increasing the repaired layer thickness from 25 mm to 50 mm. The ultimate load of corroded RC beam reduced by 17%, 10.5% and 7.4% for reference, 25 mm and 50 mm PVAFRGC repaired RC beams, respectively compared to the non-corroded RC beams. From the monitoring of the interface slip measurements during the flexural test, it was observed that there were not significant slip value and very good bond condition were achieved between the PVAFRGC repaired layer and substrate concrete.

9.2.5.2 Strengthening RC beams by NSC, SFRGC and PVAFRGC.

The effect of strengthening of RC beams by an additional layer of NSC, PVAFRGC and SFRGC on the corrosion resistance and flexural performance has been evaluated. Moreover, the effect of addition of PVAFRGC jacketing on the flexural performance have been examined. Visual inspection and steel reinforcement mass loss confirm that the beam strengthened with conventional (NSC) techniques has rust stains and longitudinal corrosion cracking in the side of the strengthening layer parallel to the corroded steel reinforcing bars. The RC beams strengthened with SFRGC and PVAFRGC additional layers did not show any localized cracks on the additional surface, rather multiple tight cracks were distributed at the additional overlay. The beams strengthened with PVAFRGC and SFRGC overlay showed a significantly reduced effect of corrosion exposure on the flexural load capacity. The flexural load of corroded RC beams strengthened with SFRGC increased by 5%, while the ultimate load of corroded RC beams strengthened with SFRGC layer were quite

similar to the non corroded specimens. On the other hand, the flexural load of corroded beams strengthened using the conventional technique by an NSC additional layer reduced by 65% compared to the non-corroded RC beams. The results also showed that the addition of reinforced NSC, SFRGC and PVAFRGC layers on the tension side increased the flexural load by about 2 times compared to the control RC beams. The strengthening of an RC beam with PVAFRGC jacketing increased the ultimate load by 50% compared to the control untreated reinforced concrete beam. The interface slip between the substrate concrete and PVAFRGC, SFRGC and NSC additional layers were recorded during flexural test. The interface slip measurements were very small for all strengthened specimens and the effect of corrosion on the interface slip was negligible.

9.3 Critical reflections and recommendations for future research.

Critical reflections;

- The major part of this research has focused on the development and experimental testing of FRGC material. As this is produced by using ternary binder geopolymer with different fibres, which produces a structurally complicated composite, for which there are no standard modelling codes, an experimental approaches was preferred (and was considered more valid) than theoretical modelling. This study has produced however, material design and mechanical characteristics that could be used in structural modelling. Theoretical models for FRGC material and the repaired and strengthened RC beams could therefore be a valuable a view for future research.
- In this study, FRGC has been evaluated in term of interfacial bond strength, differential shrinkage, curing condition, mechanical loading and durability properties (corrosion resistance). The cost of the repair material has a significant impact on the final choice of the material used for the repair and strengthening, but it should not be put before the performance characteristics. A poor choice of repair material would cause the earlier failure of the repaired structure. However, the cost of using FRGC material as repair material needs to be tied to the expected service life in order to have an adequate economic analysis of the repair.

• Fabrication of fibre reinforced geopolymer concrete for construction and strengthening applications requires a high workability and sufficient setting time, but initial strength for moving after casting also needs to be ensured. This is greatly influenced by the use of admixtures, which might need to be of a retarding type. The time taken to cast FRGC also needs to be addressed (in this PhD study, a time of 3-5 minutes was used for casting) to be applicable on an industrial scale. Thus, it is highly recommended to trial fabricating procedures in a full-scale industrial facility. Moreover, the effect of variation in environment temperature, relative humidity (RH), and volumetric size of the specimens on shrinkage behaviour of FRGC need to be address.

Suggestions for future research are given below:

Material Properties

- From the experimental results of the current study, the shrinkage of plain geopolymer showed high shrinkage values (around 2900 microstrains) and inclusion of straight steel, PVA and glass fibre reduced the shrinkage measurements to 850, 1600 and 1400 microstrains, respectively at 120 days. However, these shrinkage measurements were still high and it would be useful to investigate the shrinkage behaviour of plain geopolymer and FRGC with different type and content of shrinkage reducing admixtures.
- The shrinkage performance of FRGC evaluated by free drying shrinkage and restrained shrinkage tests are the two methods that have been used to assess the shrinkage-induced cracking of geopolymer composite. Other types of shrinkage such as plastic shrinkage, autogenous shrinkage, chemical shrinkage, creep relaxation, carbonation shrinkage, and shrinkage rate should be included in future work.
- Experimental work presented in this thesis has shown that geopolymer set under ambient temperature has better chloride and corrosion resistance compared to OPC mortar for short periods of up to 6 months. Further studies to investigate the longerterm performance of geopolymer material cast in situ for structures that are in exposed to a chloride-rich environment should be carried out.

Structural Applications

- An experimental program to investigate the reversed cyclic and dynamic loading response of FRGC material and repaired/ strengthened RC beams with FRGC.
- An experimental program to investigate the effect of different non-reinforced additional layer thickness and 3 side jacketing of FRGC on the structural performance of RC beam.
- An experimental program to investigate the durability of repaired and strengthened RC beams after freezing and thawing exposure and fire resistance.

In addition, to further improve the sustainability of geopolymer materials, it would be useful to examine the effect of inclusion of recycled fibre types such as polyethylene terephthalate (PET) fibres and Recycled Tyre Polymer Fibres on the mechanical properties and shrinkage characteristics of FRGC members. This would make use of common plastic / polymer waste materials and potentially divert these from landfill disposal.

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APPENDICES

APPENDICES 1:

PUBLICATIONS, CONFERENCES AND INVITED TALK

Journals:

- Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Cundy, Andrew and Meikle, Steve (2016). *Development of geopolymer mortar under ambient temperature for in situ applications*, Construction and Building Materials, 120. pp. 198-211. ISSN 0950-0618.
- Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Andrew, Cundy (2017) *Steel fibre reinforced geopolymer concrete (SFRGC) with improved microstructure and enhanced fibre-matrix interfacial properties* Construction and Building Materials, 139. pp. 286-307. ISSN 0950-0618.
- Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Andrew, Cundy (2017). *Tensile properties of a novel fibre reinforced geopolymer composite with enhanced strain hardening characteristics* Composite Structures, 168. pp. 402-427. ISSN 0263-8223.

Conference;

- Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Cundy, Andrew (2016). *Effect of Alkaline Activator, Water, Superplasticizer and Slag Contents on the Compressive Strength and Workability of Slag-Fly Ash Based Geopolymer Mortar Cured under Ambient Temperature* International Journal of Civil, Environmental, Structural, Construction and Architectural Engineering, 10 (3). pp. 308-312.
- Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Cundy, Andrew (2016). *Experimental investigation of the effect of silica fume on geopolymer mortar cured under ambient temperature* In: Rheological measurements on building materials 2016: Proceedings of the 25th Workshop and Colloquium, OTH Regensburg, (East Bavarian University of Technology), Regensburg, Germany, 2-3 March, 2016.
- Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Cundy, Andrew (2016). *Strength and porosity of fly ash and slag based geopolymer mortar cured under ambient temperature with variant Silica Fume forms*. Young Researchers' Forum III Innovation in Construction Materials, Imperial College London, 12th April 2016.
- Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Cundy, Andrew (2016). *Mechanical properties of steel fibre reinforced geopolymer composites cured under ambient temperature* In: Fib symposium 2016 'Performance-based approaches for concrete structures', University of Cape Town's Graduate School of Business (GSB), 21-23 November 2016.

• Al-Majidi, Mohammed Haloob, Lampropoulos, Andreas and Andrew, Cundy (2017). *Strengthening of plain concrete beams using Strain Hardening Geopolymer Composites (SHGC) layer*. In 39th IABSE Symposium Vancouver "Engineering the Future", Vancouver, Canada, 2017.

Posters presented:

- Oral presentation Brighton Doctoral College Research Student conference 2014.
- Poster presentation in Geopolymer concrete for ENSOR workshop/ University of Brighton.
- Poster presentation Brighton Doctoral College Research Student conference 2015

Academic Activity and invited talks;

- Attendance to Making Concrete Greener conference on 17 April 2015/ SCI/ London, UK.
- Attendance to 44th Annual Convention Symposium. The Future of Precast An opportunity to hear how developments are being applied in Precast Concrete. Loughborough University. 14th April 2016.
- PhD seminar of 'Sustainability and Resilience Engineering (SuRE)' research group, 18th May 2016. University Of Brighton.
- Attendance to 8th Geopolymer Camp 2016, at the University of Picardie, Geopolymer institute, Campus of Saint-Quentin, North of Paris, France, on July 4-6, 2016.
- Attendance to 44th Annual Convention Symposium. Alkali-activated cementitious material and concrete. The Institute of Concrete Technology and University College London. 21 September 2016.