



**Development of Lightweight Geopolymeric Materials
using Coal Fly Ash and WastePaper**

By

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Declaration

I, **Natali Maruva Sharlene Nyabanga**, declare that the contents of this thesis represent my own unaided work and that the thesis has not previously been submitted for academic examination towards any qualification. Furthermore, it represents my own opinions and not necessarily those of the Cape Peninsula University of Technology.

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Date

Abstract

The increase in population results in the intensification of coal combustion for power generation. The exponential population growth in South Africa results in an increase in unused process by-products such as coal fly ash (CFA) and wastepaper. In power plants, by-products such as fly ash is extracted from the exhaust gases and disposed to landfills. The demand for and consumption of paper in daily life is increasing every year, leading to paper disposal problems. Waste paper has drawn interest from researchers as a reinforcement material for geopolymer due to its properties such as lightweight, affordability, availability, and toughness.

Non-load bearing materials are currently manufactured from gypsum and wood, but these materials are relatively expensive due to the additional costs incurred by the addition of non-flammable constituents. CFA and waste paper which are currently causing disposal problems might provide an alternative and cost-effective route to manufacturing non-flammable, lightweight, low cost and high strength non-load bearing geopolymeric materials. The chief aim of this study is to develop lightweight, high strength geopolymeric material using waste materials such as waste paper and CFA. Specifically, the objectives of this study are to evaluate the compressive strength, weight, tensile strength, water absorption and shrinkage of the reinforced geopolymer synthesized using different characterization techniques.

In addition, the impact of waste paper on CFA ratio, NaOH molarity and curing temperature on compressive strength, geopolymer weight, water absorption and shrinkage properties will be investigated in order to determine the optimum conditions for the synthesis of reinforced geopolymers. Characterization of the geopolymer by x-ray diffraction, x-ray fluorescence, and electron microscopy scanning were included in this analysis. This study will lead to the development of cheap, lightweight, high strength construction material by making use of waste paper and fly ash which is currently constituting an environmental nuisance. The result of this research could find application in the construction industries.

No aggregates, sand or cement were used in any of the formulations developed in this study. Waste paper content was varied from 10 % to 40 %. The optimum waste paper content was found to be 20 % as the geopolymer weight was 0.856 kg, compressive strength between 3.8 MPa and 4.5 MPa; the water absorbed after 30 minutes of immersion was 0.360 L and the

shrinkage percentage was found to be between 18 % and 20 %. The geopolymer product had a mass varying from 0.91 to 1.2 kg per 100 mm³ and density of 600 - 1000 kg/m³. The operating cost was calculated, and it was determined that for a 5 000 kg/year production, one geopolymer costs ZAR 31.70 and at 160 000 kg/year the block costs about ZAR 5.02.

The optimum formulation used consisted of 2.4 kg of CFA, 0.6 kg of WP, 1.80 kg of H₂O, 0.64 kg of Na₂SiO₃ and 0.32 kg of NaOH. The mixture was mixed for a total time of 45 minutes and the hydraulic pressure used for moulding was 5 MPa. The geopolymer was cured for less than 60 hours and aged for 7 days prior to any tests. Partitioning wallboard manufactured using this formulation weighed 35 kg/m² and the lightweight brick manufactured weighed 1.1 kg at 220 mm in length, 110 mm in width and 49 mm in depth. It was concluded that the materials can be used for non-load bearing construction material and should be installed at 1 m above ground level to avoid moisture absorption from the ground.

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Dedication

This thesis is dedicated to my loving late father

Christopher N. Nyabanga

For all the love and sacrifices you made to ensure I have a better and brighter future,
I am forever grateful to you Daddy. You are forever missed.

List of Publications and Presentations

Publications

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List of Symbols

Scientific Symbols

Symbol	Description	Units
Q	Capacity	Ah/Wh
I	Current	A
E	Energy	J
η	Energy Efficiency	%
M	Mass	kg
M_r	Molar Mass	kg/kmol
N	Moles	kmol
P	Power	W
R	Resistance	Ω
V	Voltage	V

List of Symbols

Chemical Specie Symbols

Specie	Description
NaOH	Sodium Hydroxide
Na ₂ SiO ₃	Boric Acid
Cd	Cadmium
CaO	Calcium Oxide
H ₂	Hydrogen
Si	Silicon
Al	Aluminum
O	Oxygen
Fe	Iron
Ca	Calcium
FeS	Iron Sulphide
Ni	Nickel
S	Sulphur
Ti	Titanium
H ₂ O	Water
Cd	Cadmium
B	Boron
H ₂ O	Water
Zn	Zinc

List of Abbreviations

CFA	Coal Fly Ash
HGC	High-Grade Coal
LGC	Low-Grade Coal
LWG	Light Weight Geopolymeric
LWGM	Light Weight Geopolymeric Material
WP	Waste Paper
WPP	Waste Paper Pulp
FRG	Fibre Reinforced Geopolymer
GFRC	Glass Fibre Reinforced Concrete
RCA	Recycled Concrete Aggregate
WSA	Waste Paper Sludge Ash
GBFS	Granulated Blast Furnace Slag
LOI	Loss of Ignition
RPM	Revolutions Per Minute
SEM	Scanning Electron Microscopy
XRF	X-Ray Fluorescence
XRD	X-Ray Diffraction
ASTM	American Society for Testing and Materials

Chapter 1: Introduction

1.1. Background

The main source of power generation around the world is coal (Coal & electricity, 2019). The increase in population results in the intensification of coal combustion for power generation (Coal & electricity, 2019). It has been reported by Ahmaruzzaman (2010) that an estimate of 600 million tonnes of coal ash is being produced annually worldwide. The electricity generated from coal accounted for about 30 % and it is estimated to increase to 46 % by the year 2030 (Yao et al., 2015). China uses 50% of the world's coal, being the top of the list globally while South Africa comes 6th in the world, using approximately 2.4% of world coal consumption (Yao et al., 2015). South Africa depends mainly on coal for power generation with approximately 85% of its power from coal and 15% from hydroelectric, gas and nuclear power (Stone, 2015).

Coal is categorized into two groups; namely low-grade coal (LGC) and high-grade coal (HGC). HGC contains fewer inorganic minerals, consisting mainly of clays, calcite, dolomite, pyrites and silica, whereas LGC has a high inorganic mineral content (Ahmaruzzaman, 2010). South African power plants burn LGC in electricity generation. LGC produces several by-products/mineral wastes, one of which is known as coal fly ash (CFA) (Petrik et al., 2003). Coal combustion produces by-products, namely bottom ash and coal fly ash. The latter is extracted by electrostatic precipitators/bag filters from exhaust gases in power plants and disposed to landfills. Sibanda et al. (2016) reported that about 40 million tonnes of coal fly ash were being produced in South Africa annually. The constituents of CFA, glassy particles contain mainly Si, Al, O, Fe and Ca and traces of toxic elements (Musyoka et al., 2012; Inada et al., 2005; Querol et al., 2002).

The disposal of CFA is becoming a challenge in power stations. CFA particles are highly polluting due to their enrichment with potentially toxic trace elements that are condensed by flue gas (Ahmaruzzaman, 2010). CFA consists of glassy particles that are prone to wind distribution and therefore may cause air pollution which further affect human health by causing airborne diseases such as asthma, silicosis and bronchitis (Madzivire et al., 2010). When the CFA is dumped in landfills, it has the potential to leach into the soil, contaminating surface and ground waters (Heebink and Hassett, 2001).

A considerable amount of research has been done in order to minimise the increasing toxic threat posed by CFA to the environment. Subsequently, an economically viable solution to this problem would include using waste materials for new products instead of land disposal (Muriithi et al., 2013; Mainganye, 2012; Querol et al., 2002; Swanepoel & Strydom, 2002). Zhuang *et al.*, (2016) has reported that CFA can be used for many applications such as an adsorbent for the treatment of acid mine water, zeolite manufacturing, construction applications, lightweight aggregates, and recently it has been mostly used to make geopolymer. Davidovits first applied the term geopolymer to three-dimensional aluminosilicate alkali binders produced by the activation of aluminosilicate alkali silicate materials (Duxson et al., 2005).

Geopolymers can display a wide range of properties and characteristics, including high compressive strength, low shrinkage, fast or slow setting, lightweight, acid resistance, fire resistance, and low thermal conductivity, depending on the selection and processing conditions of the raw material. (Davidovits., 2008; Duxson et al., 2006; Singh et al., 2015). A significant amount of research has been done on geopolymer synthesis and its possible applications. The mechanical properties of a geopolymer determine its application in a specific industry. Geopolymers can range from load-bearing construction materials such as concrete, bricks and roof tiles as compared to non-load-bearing materials such as drywall and indoor furniture (Duxson et al., 2006; Duxson & Van Deventer, 2009; Abdullah et al., 2012; Ricciotti et al., 2017).

Many ways have been discovered to improve the properties and characteristics of geopolymers. Most researchers have looked at the weight of the geopolymer, the compressive strength, fire resistance characteristics, flexibility and ductility (Shaikh, 2013; Duxson et al., 2006). CFA has been mixed with many fibres such as rice husks, plastics, clay, sand and foam for the purpose of improving geopolymer properties. Waste paper has drawn interest to researchers as a reinforcement material for geopolymer due to its properties such as low density, cost-effectiveness, stiffness, high filling levels possible, energy conservation, desirable strength to weight ratio and high availability (Dachowski & Kostrzewa, 2016; Aliabdo et al., 2016). The demand for and consumption of paper in daily life is increasing every year, leading to paper disposal problems (Paridah et al., 2015).

Waste paper is typically disposed of and often burned on open dump landfills, contributing to environmental problems such as dust, soil, surface and groundwater pollution caused by leachate and greenhouse gas emissions induced by the waste decomposition process (Okeyinka & Idowu., 2014; Tisserant et al., 2017). Waste paper has been utilised for purposes such as fibre cement board (Ashori et al., 2011), masonry blocks (Modry, 2001; Fuller et al., 2006a; Akinwumi et al., 2014), low density board (Esmeralda et al., 2000), papercrete (Fuller et al., 2006a; Fuller et al 2006b), brick (Jegatheeswaran, 2011), and plastering mortar (Aciu et al., 2014). As wastepaper has been used extensively in the production of lightweight construction material, its application as a lightweight geopolymer filler in this report is viable.

1.2. Research Problem Statement

The notable demand for electricity has increased the consumption of coal which proportionally increases CFA production. CFA pollutes air and groundwater, causing serious health and environmental problems. As the population continues to increase, waste generation continues to increase, and without a proper plan to limit waste generation, the increase in waste continues. Looking at the current rate at which mass solid waste is being produced, if acceptable measures to mitigate waste generation are not in place, the occurrence of a tsunami of waste is predictable. It is therefore important to look at the beneficiation of these wastes/by-products in various industries.

It is therefore advisable to replace conventional building materials such as cement with different materials, preferably waste materials, because cement and aggregates used in building are increasing in cost. On the other hand, a continuous increase in the global population has resulted in a demand for waste paper products. It is therefore advisable to implement and promote the recycling of paper globally. Only a small percentage of waste materials are being recycled. Most waste materials are disposed of in open spaces as litter. The poor disposal of litter results in pollution of land and water, as well as clogged drains which increases the risk of flooding.

Non-load bearing materials are currently manufactured from gypsum and plaster of Paris, but these materials are relatively expensive due to additional costs incurred by the addition of non-flammable constituents. CFA and waste paper which are currently causing disposal problems

might provide an alternative and cost-effective route of manufacturing non-flammable, lightweight, low cost and high strength non-load bearing geopolymeric materials.

1.3. Aims and Objectives

In general terms, the chief aim of this study is to establish optimum conditions production of a lightweight, high strength geopolymer using waste materials such as CFA and waste cardboard (WP). This will be achieved by the following objectives of the study as listed below:

- Investigate the effect of WP to CFA ratio, NaOH molarity and curing temperature on compressive strength,
- Investigate the effect of WP to CFA ratio, NaOH molarity and curing temperature on geopolymer weight,
- Investigate the effect of WP to CFA ratio, NaOH molarity and curing temperature on water absorption.
- Evaluation of production cost of non-load bearing construction geopolymeric materials.

1.4. Significance of Research

This study aims to develop a lightweight geopolymer using waste cardboard as an aggregate. The total use of CFA and waste cardboard in the development of construction materials could be a turning point for the paper recycling industry and construction industry. Reducing, reusing and recycling waste materials can bring about the benefit of significant conservation of natural resources. This research will be focused on the development of eco-friendly, non-load bearing building materials. This study will contribute positively to the environment because the main materials used are currently causing environmental problems due to inadequate disposal. This will result in more available land which could be used for housing and school developments.

1.5. Research Questions

The objectives listed above should be achieved by answering research questions listed below:

- What is the relationship between the WP: CFA ratio to compressive strength?
- What is the effect of NaOH concentration, water content and curing temperature on compressive strength and shrinkage of a lightweight geopolymer?
- How is water absorption affected by the waste cardboard content?
- What is the optimum mix and curing conditions for a lightweight geopolymer?
- Where in the construction industry can the lightweight geopolymer be used?

1.6. Delineations

The waste paper used for the composite board can range from cardboard to office paper; however, this study focuses on waste paper from cardboard boxes only. The CFA used was from a South African power station and class F will be used for the development of the geopolymer due to its availability.

1.7. Research Approach

The research approach to attain the objectives of the study is as follows:

Literature Review

The literature review was done extensively to determine the gaps in research with reference to the manufacturing of lightweight geopolymeric materials using coal fly ash and waste paper.

Experimental work was used to:

- i. Determine the optimum mix composition for the lightweight geopolymer material (LWGM),
- ii. Evaluate engineering properties of LWGM according to relevant standards.

1.8. Outline and structure of the thesis

This thesis comprises 6 chapters. The structure of this study is defined briefly in each of the following chapter headings:

Chapter 1 - Introduction:

The introduction chapter provides insight into the problems and purpose of the study with reference to knowledge and information that already exists on geopolymeric materials. Once the background is presented, the aims and objectives of the current report are clearly stated followed by the significance of the current research, the research questions. and delimitations of research.

Chapter 2 - Literature Review:

Chapter 2 contains the literature study. The purpose of this chapter is to understand, and familiarise the researcher with, an in-depth theoretical background and extensive engineering principles applied in this research. The literature review chapter consists of the fundamental background of the development of geopolymeric material, in-depth geopolymer formation as well as properties of reinforced geopolymers.

Chapter 3 – Methodology:

The experimental methodology chapter describes the method used for all experimental work, test procedures, equipment used and programmes in detail. This chapter describes and clarifies how the materials discussed in the literature review were employed in practice to solve the research problem.

Chapter 4 – Experimental Results and Discussion

The purpose of this section is to analyse and discuss all the results obtained from Chapter 3. The numerous empirical data-sets were analysed and discussed in this chapter. Mass and energy balance calculations were done and discussed in this chapter too. The results were evaluated and assessed to evaluate the objectives set in Chapter 1.

Chapter 5 – Mass and Energy Balance and Costing Evaluation:

One of the main aims of this study is to develop a cost-effective method and product, therefore this chapter serves to determine whether the process of manufacturing and the end product is indeed cost-effective.

Chapter 6 – Conclusion and Recommendation:

This chapter concludes and summarises all the results presented in Chapters 4 and 5. Chapter 6 also provides recommendations for further studies of the research work in lightweight construction materials using waste materials coal fly ash and waste paper.

Chapter 2: Literature Review

2.1. Overview

Chapter 2 focuses on the comprehensive review of geopolymers, geopolymeric formation and reinforced geopolymeric construction materials. CFA characteristics, environmental impacts, and applications are discussed in section 2.2 to section 2.4. A background on geopolymerisation formation, effects of processing parameters and applications is given in section 2.5 to 2.7. Reinforcement materials and their effects in geopolymers are discussed in section 2.8. The detailed background on waste paper as a filler/aggregate/reinforcement material is described in section 2.9. Properties of geopolymers are discussed in section 2.10 and the current status of the project are assessed in section, 2.11 followed by the summary of the chapter.

2.2. Coal Fly Ash

Coal fly ash (CFA) consists of dense traces of fine particles collected by electrostatic precipitators or other particle filtration devices from flue gases before the flue gas enters the chimneys (Zhuang et al., 2016). Coal fly ash is primarily grey in colour, abrasive in nature, somewhat alkaline and refractory (Ahmaruzzaman, 2010). It is comprised mainly of silicon and aluminium and is thus classified as a pozzolan, and can form cementitious products when mixed with water and calcium hydroxide (Ahmaruzzaman, 2010). CFA is generated from exhaust gases at 1200 to 1700°C and its constituents depend on the feed coal; as a result, CFA is one of the most complicated materials to be characterised (Blissett & Rowson, 2012). Coal fly ash causes environmental and health problems due to its constituent trace elements such as As, Pb, Sb, Ba, V, etc. (Du Plessis et al., 2014).

South African power plants, including Eskom plants, generate about 40 million tons of CFA every year (Sibanda et al., 2016), and from this amount, only approximately 5.5% is utilized effectively in the construction industry, while the rest is stockpiled (Petrik, 2004). According to the latest available report from Eskom (2016), the annual production of Eskom CFA is 32 million tonnes per annum of which only approximately 8% is reused, the remaining 92% being disposed in ash dumps. Traditionally, CFA has been disposed of in landfills or ash lagoons; however, these lagoons have been known to breach and cause significant harm to the local

communities surrounding them (Blissett and Rowson, 2012). Because of the presence of soluble metal products, poor management of this waste material has led to serious environmental concerns, including possible pollution of land, surface and groundwater.

2.3. Properties of Coal Fly Ash

The main focus of this section is to analyse coal fly ash and establish the chemical and physical compositions and behaviour of coal fly ash in various applications.

2.3.1. Physical Properties

The morphology or physical characteristics of coal fly ash are influenced by the feed coal's combustion temperature and the power plant's cooling cycle (Yao et al., 2015). It was deduced that CFA consists of solid and cenospheres as shown in a scanning electron microscope (SEM) figure 2.3-1 . Analysis of X-ray diffraction has also been used to test fly ash morphology and CFA includes quartz and mullite aggregates (Blissett & Rowson, 2012). The colour of CFA can be between grey and black and this depends on the amount of burnt carbon in the coal fly ash. The specific gravity of CFA is between 2.1 and 3 with a high specific surface area of 1000 m²/kg which allows it to be used in applications that need adsorption. In the fly ash, the carbonaceous material is made up of small crystals. Many bituminous CFA particles' size distribution is generally similar to silt (less than 0.075 mm or No. 200 sieve). Although sub-bituminous CFA is also silt-sized, it is usually slightly coarser than bituminous CFA (Ahmaruzzaman, 2010; Yao et al., 2015).

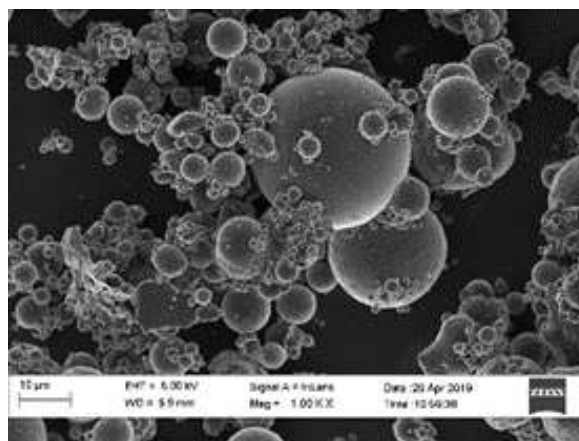


Figure 2.3-1: SEM images of Matla CFA

2.3.2. Chemical Properties

The thermal process for the conversion of coal into fly ash (combustion, gasification) and the properties of the coal itself (bituminous e.t.c) have a major impact on fly ash's chemical properties (Musyoka, 2012; Madzivire et al., 2010). There are essentially four types of coal, including anthracite, bituminous and sub-bituminous coal, and lignite, with heating nature, chemical composition, ash content, and geological origin differing from each. Silica, alumina, iron oxide, and calcium, with varying amounts of sulphur, are the main components of bituminous coal fly ash. Lignite and sub-bituminous carbon fly ash is characterized by higher calcium and magnesium oxide amounts and decreased silica and iron oxide levels as well as lower carbon content relative to bituminous CFA. For service boilers, anthracite is burnt, so only small amounts of anthracite coal fly ash are present. (Ahmaruzzaman, 2010; Blissett & Rowson, 2012).

Depending on their calcium oxide content, CFA can be graded as Class C or Class F. Class C (cementitious and pozzolanic) and Class F (pozzolanic) are differentiated in detail by Eisele et al. (2004). Class C fly ash is high in calcium and is produced mainly from the burning of lignite coal sources. Class C fly ash has a cumulative concentration of SiO_2 , Al_2O_3 and Fe_2O_3 between 50 wt. % and 70 wt. % and more than 20 wt. % content of CaO. Class F fly ash is low in calcium and is created by burning anthracite or bituminous coal. Type F fly ash has a minimum content of more than 70% SiO_2 , Al_2O_3 and Fe_2O_3 and a content of less than 10% CaO (Andini et al., 2008; Izquierdo et al., 2009).

Class F and Class C CFA have major difference in the amount of calcium and the level of silica, alumina and iron. In Class F fly ash, total calcium in association with silica and alumina usually varies from 1 to 12 %, mostly in the form of calcium hydroxide, calcium sulfate, and glassy components. Class C fly ash, on the other hand, may have registered a calcium oxide content of up to 30–40 %. Another distinction between Class F and Class C is that in Class C fly ash, the number of alkalis (combined sodium and potassium) and sulphates (SO_4) is usually higher than in Class F fly ash (Ahmaruzzaman, 2010).

Table 2.3-1: Chemical composition of different CFA. Adopted from Ahmaruzzaman (2010).

Component (wt.%)	Bituminous	Sub-bituminous	Lignite
SiO ₂	20–60	40–60	15–45
Al ₂ O ₃	5–35	20–30	10–25
Fe ₂ O ₃	10–40	4–10	4–15
CaO	1–12	5–30	15–40
MgO	0–5	1–6	3–10
SO ₃	0–4	0–2	0–10
Na ₂ O	0–4	0–2	0–6
K ₂ O	0–3	0–4	0–4
LOI	0–15	0–3	0–5

2.3.3. Coal Fly Ash Mineralogy

X-ray diffraction (XRD) analysis machine will determine the mineralogical composition of fly ash, which is based on the geological factors related to the formation and deposition of coal and its combustion conditions. Quartz, kaolinite, illite, and siderite are the primary mineral types. In the unreacted coals, the less prevalent minerals are calcite, pyrite, and hematite. The main crystalline components of low-calcium ash are quartz and mullite, while high-calcium fly ash consists of quartz, C₄AS, and C₃A, etc. Glass, mullite, quartz, char, hematite-magnetite, anhydrite-gypsum, feldspar, lime-portlandite, clay and mica minerals, cristobalite-tridymite, calcite-ankerite, corundum, jarosite, and some Ca and CaMg silicates are the main phases present in coal fly ash (Vassilev & Vassileva, 2007; Blissett & Rowson, 2012). Quartz, kaolinite, elite and mullite with trace amounts of magnetite and hematite phases are the dominant minerals in FA (Ahmaruzzaman, 2010; Moreno et al., 2001; Musyoka, 2009). Mullite and quartz are the major crystalline phases found in FA. The amorphous glassy phase in CFA is responsible for its pozzolanic nature (Ahmaruzzaman, 2010; Querol et al., 2001; Fisher et al., 1978). In the stringency of the properties needed in the CFA for its productive use, the several distinct end uses of fly ash differ considerably.

2.3.4. Environmental Problems

The impact that CFA has on the environment has been recognized fully due to the increasing/high demand for electricity. The most often used technique for disposing of CFA is dumping it over vast landfills, which results in human beings and the environment being endangered (Zhang, 2013; Querol et al., 2002). In South Africa, Eskom power generation stations produce about 40 Mt of CFA annually (Sibanda et al., 2016; Babajide et al., 2010), and only a small percentage of the ash is utilised efficiently in the building sector for the production of cement and brick. Unused CFA is pumped into large ash landfills via pipes in watery sludge form. (Ahmaruzzaman, 2010; Petrik, 2004; Van Hamburg et al., 2004).

Coal fly ash contains small glassy particles that escape into the atmosphere and cause various health problems. Inhalation of these particles can cause nose, throat and respiratory tract irritation (Yao et al., 2015). If there is serious exposure or continuous exposure to CFA, it can cause silicosis, asthma and other respiratory tract diseases. There have been other cases reported of gastrointestinal illness, birth defects, and impaired bone growth in children (Bhanarkar et al., 2008; Goodarzi, 2006). Fly ash can even enter the subsoil, inducing siltation, disrupting natural drainage systems, and contaminating the environment with toxic metals.

CFA contains 4 to 10 times more trace metals than the parent coal and it has been seen as one of the major sources of harmful components released into the atmosphere (Patra et al., 2012). Metal pollutants such as V, Cr, Ni, Cd, and Pb have been documented to pose potential environmental hazards at low levels (Patra et al., 2012). Nyale et al. (2014) studied the leaching activity and geochemical partitioning of trace elements, such as As, Zn, Pb, Ni, Mo, Cr and Cu, in a 20-year-old fly ash landfill. The trace elements leached quickly from the discarded CFA and therefore posed a threat to soil and crop production. Although some elements found in CFA are deemed beneficial for the growth of plants, these benefits are outweighed by the considerable levels of environmental concerns associated with CFA, due to its constituent toxic elements (Yao et al., 2015).

Upon coal combustion, the bulk of U, Th and their decay products are extracted from the initial coal matrix and are dispersed between the gas phase and the solid combustion products. The determination of exposure to radiation from fly ash is highly dependent on the accumulation of radioactive elements in the parent coal and fly ash left after combustion. Mathur et al. (2008)

examined the rate of Rn exhalation in fly ash samples in India and found a rise in radionuclide emission following coal combustion.

2.4.Applications of Coal Fly Ash

Section 2.4 discusses several CFA applications. Dumping CFA in landfills has been an issue over many years due to the environment and health issues. Re-utilisation of coal fly ash is therefore encouraged as this option benefits the environment and individuals in a positive way. Firstly, looking at the environment, the disposal area is minimised, allowing other uses for land such as agriculture and housing. Secondly, disposal costs are minimized. Thirdly, the sale of the by-product will bring financial returns. Fourthly, reutilising CFA will substitute for certain costly natural resources. Such methods and applications include, but are not limited to, cement and concrete buildings, roadways and pavements, construction materials as a lightweight aggregate, irrigation and water treatment, and the environment. The following figure 2.4-1 is a short summary of each of the alternative uses of fly ash and the following sections 2.4.1 to 2.4.8 will describe briefly the related work that has been carried out and how it applies to each alternative usage.

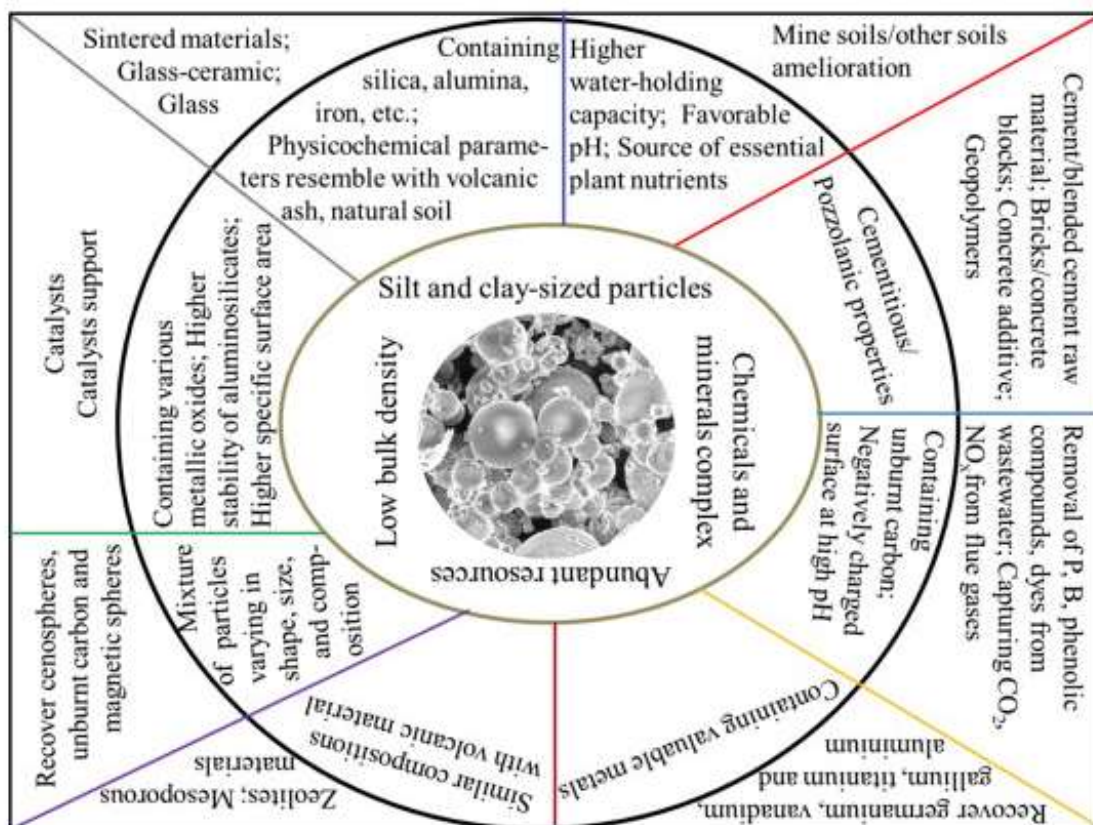


Figure 2.4-2: Different applications of CFA. Adopted from Yao et al. (2015).

Because of its high SiO_2 and Al_2O_3 material, CFA has found application in zeolite and geopolymer synthesis (Querol et al., 2002). In recent times, the synthesis of zeolites using CFA has drawn more attention, due to the fact that CFA is an inexpensive source of silica and alumina and in addition, there is an increasing number of studies on CFA because zeolites have wide applications and they have several economic benefits (Musyoka, 2009). Swanepoel and Strydom (2002) studied the similarity of CFA to pozzolans and has encouraged the use of CFA in the synthesis of geopolymers, a material which consists of a polymeric Si–O–Al framework (Nyale et al., 2013; Swanepoel and Strydom, 2002).

2.4.1. Environmental Waste Removal

Adsorption is one of the promising flue gas capture technologies for CO_2 , SO_2 and NO_x . The carbon particles in carbon fly ash boost the carbon fly ash surface area, rendering it an adsorbent. To further boost adsorption efficiency, the carbon can be activated. Lu & Do (1991) and Rubio & Izquierdo (2013) used fly ash to extract NO_x . Izquierdo & Rubio, (2008) measured CFA as an SO_2 reduction adsorbent. Some samples showed a good ability to remove SO_2 , which could be due to their textural properties. Sarmah et al. (2013) and Wilfong et al. (2016) investigated the capture potential of composites based on amine fly-ash. A study of material cost and ability for adsorption showed that they were cheap and efficient in trapping CO_2 (Muriithi et al., 2013).

2.4.2. Wastewater treatment

There has been extensive work on the use of fly ash as an adsorbent for water treatment. The use of fly ash as an adsorbent for removing Cu_2^+ from chemical wastewater was studied by Panday et al. (1985). Fly ash is used to remove various metal ions including Pb, Zn, Mn, Cd, Cr, and Ni and As and B. (Visa et al., 2012; Koshy & Singh, 2016; Ayanda et al., 2014)

2.4.3. Catalysis

Metal and metal oxide catalysts are widely used in various industries as catalysts to speed up reactions. CFA mainly consists of various metal oxides with higher thermal stability and iron oxides. Utilising the waste material (CFA) for heterogenous catalysis is beneficial for the environment as well as economical (Wang, 2008). In addition, fly ash can also be used as

catalyst supports for various reactions due to the higher stability of its main constituent aluminosilicates. Research has been done on the use of CFA as catalysts and it was discovered CFA can also be used as catalyst support. For the Knoevenagel condensation reaction, CFA-based CaO was used as a recyclable solid base catalyst (Jain et al., 2011). Chakraborty et al. (2010) prepared a CaO catalyst aided by CFA for soybean oil transesterification. Babajide et al. (2010) used CFA as a fuel for biodiesel processing. By loading sulphated zirconia on chemically activated fly ash, Khatri et al. (2010) synthesized a solid acid catalyst for benzylation reactions (Saputra et al., 2012; Mostafa Hosseini et al., 2014; Vichaphund et al., 2018).

2.4.4. Recovery of rare earth elements

Recent studies have been investigating the extraction of rare earth elements from coal fly ash (Seredin & Dai, 2012; Kashiwakura et al., 2013; Eze, 2014; Franus et al., 2015). Rare earth elements are a group of elements recognized as lanthanides plus yttrium and scandium and are used in emerging technologies such as green energy systems, hi-capacity fuel cells, wind turbine magnets, solar cells, and they are also used by the aviation industry (Franus et al., 2015).

2.4.5. Agriculture

CFA may be used for soil enhancement in the agricultural industry due to its efficiency and effectiveness in promoting soil fertility and crop development. The high proportion of elements such as Na, Zn, Ca, K, Mg and Fe increases harvests of different crops. Fly ash has tremendous potential in farming due to its overall effectiveness in soil health and crop yield modification. The use of fly ash in agriculture is limited compared to other industries. (Bau et al., 2009; Ahmaruzzaman, 2010; Shaheen et al., 2014). Shaheen et al. (2014) and Ukwattage et al. (2013) reported the applications and benefits of the use of CFA in agriculture, including water absorption, soil water holding power, hydraulic conductivity, and bulk soil accumulation for growth. Basu et al. (2009) further noted that the use of a large amount of acidic CFA on acidic soil causes phytotoxicity as it can introduce toxic metals, and therefore needs to be applied with care (Lee et al., 2008).

2.4.6. Construction Industry

A substantial amount of research on coal fly ash utilisation in the construction industry has been conducted for more than 3 decades. CFA, often used in cement aggregates, water, and other additives, creates a concrete blend that is well tailored to road, airport runway and bridge construction (Motz & hostageer, 2001). Essentially, there are three uses for cement-based CFA including cement substitution in Portland cement, as it lowers carbon emissions, pozzolanic content in the manufacture of pozzolanic cement, and cement retardant as a substitute for gypsum (Ahmaruzzaman, 2010).

CFA concrete has several significant advantages, is more durable and yet less costly than other conventional Portland cement blends. The use of CFA in concrete increases its workability and is commonly promoted as a partial substitute for cement. This also cuts construction costs. CFA concrete provides the steel with a very strong and stable protection against natural weathering. The use of coal fly is likely to grow further in the construction industry. CFA is often used in the manufacture of tiles, lightweight aggregates for mortars and manicure plates, fracturing materials and additives for the ceramic industry (Querol et al., 2002; Yue & Chen, 2013; Ntuli et al., 2017).

2.4.7. Synthesis of Zeolite

Zeolites are microporous aluminosilicate minerals widely used as adsorbents and catalysts owing to their crystalline nature and the abundant AlO_4 . Zeolites occur naturally but can also be artificially synthesised (Hu et al., 2017). CFA has similar composition to volcanic material from where zeolites are extracted, therefore CFA gained popularity due to these properties (Ahmaruzzaman, 2010; Vichaphund et al., 2014). Zeolite synthesis from CFA has been one of the most researched sectors on applications of CFA around the globe ever since the groundbreaking work of Holler & Wirsching (1985). Different types of CFA-based zeolites have been made over the decades.

Zeolite synthesis is traditionally produced by hydrothermal crystallization under alkaline factors (Querol et al., 2002). Three phases, i.e. dissolution, condensation and crystallization, occur in alkali hydrothermal reactions for zeolite production (Murayama et al., 2002). Synthesized zeolites are used for various engineering and agricultural applications for water purification,

gas cleaning and soil alteration (Wang et al., 2009; Ahmaruzzaman, 2010; Vichaphund et al., 2014)

2.4.8. Synthesis of Geopolymers

There has been significant research around the world into the development of geopolymers from CFA since Joseph Davidovits introduced the technology first known as geopolymerisation in the 1970s, which involves the alkali activation of aluminosilicates, and termed the resulting product 'geopolymer' (Davidovits, 2002; Dimas et al., 2009; Jha & Budhamagar, 2013; Nazari & Sanjayan, 2015; Kaur et al., 2018). Geopolymer materials can be utilised for various purposes in the construction industry to replace cement and concrete resin and binders, and in arts and archaeology (Nyale et al., 2013; Shaikh, 2013; Colangelo et al., 2013; Geopolymer.org, 2018).

2.5. Geopolymers

Geopolymer is a hybrid three-dimensional alkali aluminosilicate substance formed by the interaction of a solid aluminosilicate with a concentrated aqueous alkali hydroxide or silicate solution. Another concept used in geopolymers is inorganic polymers (Blissett and Rowson, 2012; Duxson et al., 2006; Davidovits, 1988). Geopolymers are chains with amorphous nature at ambient to medium temperatures and crystalline at temperatures above 500 °C (Mackenzie & Welter, 2014; Nyale et al., 2013; Duxson et al., 2006; Bamkwocki et al., 2004). Research in geopolymers has increased over the years, as both the ceramic and bonding industries will benefit greatly from the implementation of new technology for the manufacture of geopolymer-based products (Ciofi et al., 2003).

2.5.1. Geopolymer Formation

Since the development of geopolymers by Joseph Davidovits in 1979, several aluminosilicates are used for geopolymerisation, which include feldspar, kaolinite, including industrial solid deposits such as CFA, metallurgical slag, wastes from mines, etc. In this thesis coal fly ash is used. Geopolymerisation is caused by the chemical composition, mineral composition, morphology, fineness and glass phase content of aluminosilicate (Kumar et al., 2015; Wattimena et al., 2017). The main criteria for producing stable geopolymers are that the source materials should be extremely amorphous and have ample reactive glass content, low demand for water and be able to release aluminum quickly (Singh et al., 2015).

Geopolymerisation formation is not entirely understood, therefore, it is usually described as a three step process. Dissolution is the 1st step followed by reconstruction and polymerisation. The type of aluminosilicate, solid to alkali silicate ratio, water content, reaction condition and mixing time affect the kinetics of the geopolymerisation formation (Nyale et al., 2013).

Figure 2.5-1 shows a diagram of the geopolymer formation step process.

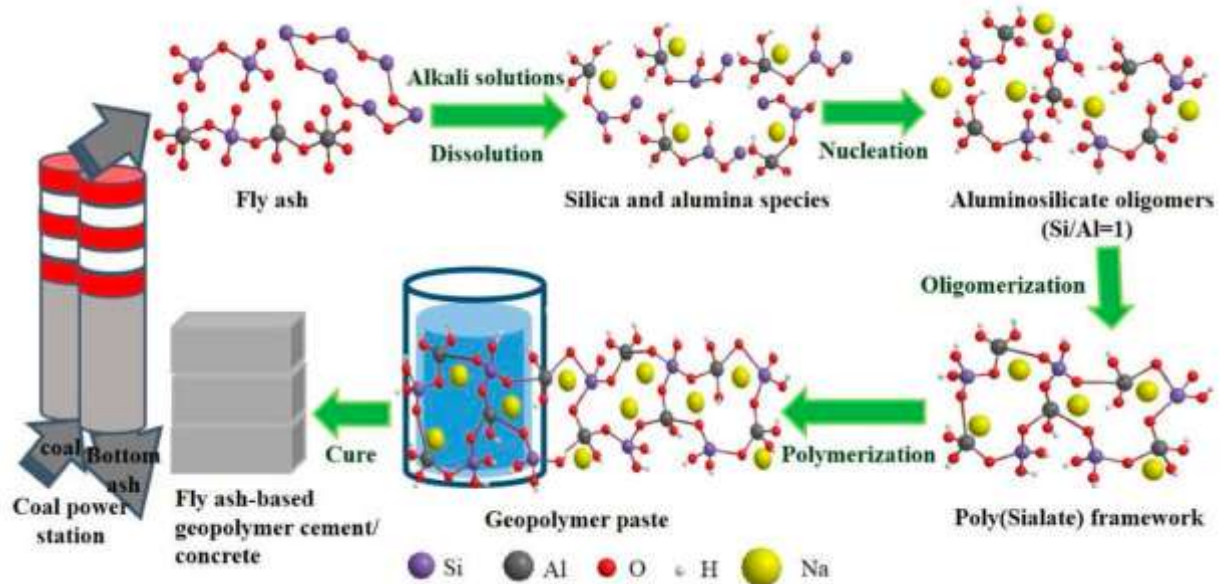


Figure 2.5-1: The process from CFA to CFA-based geopolymer. Adapted from Zhuang et al. (2016).

i. Dissolution/Hydrolysis

The production of aluminate and silicate species mechanism is produced by the dissolution of the solid aluminosilicate source by consuming water. The beginning of the dissolution stage is when hydroxide ions break the silica and the alumina bonds from the aluminosilicate source (CFA). The dissolution of aluminosilicates precursors from the surface of the material follows immediately after hydrolysis. In high alkaline conditions, the geopolymerisation formation takes place when aluminosilicates are dispersed in the aluminate and silicate tetrahedral units (Duxson et al., 2006; Sindhunata, 2006; Abdullah et al., 2012; Nyale et al., 2013; Singh et al., 2015).

ii. Restructuring

In the solution, the aluminosilicate precursors are mobile and are able to partially orientate themselves to obtain a more thermodynamically stable state before they gel through the polycondensation mechanism (Nyale et al., 2013). The dissolution and restructuring steps are highly dependent on thermodynamic and kinetic parameters (Komnitsas & Zaharaki, 2007).

iii. Polymerisation

At high pH levels the dissolution rate of aluminosilicate increases and rapidly creates a supersaturated aluminosilicate solution. The aforementioned is achieved when the saturated concentrations are lower than the dissolved species. Once there is complete saturation, the

polymerisation of Al^{3+} and Si^{4+} is attained. Extrusion of water that was consumed during dissolution occurs and shrinkage of material is observed. During the polymerisation stage oligomers form large networks in a gel or aqueous phase. The system continues to rearrange and reorganize after gelation stage, increasing the connectivity of the gel network. The continuous increase of the network results in a three dimensional aluminosilicate network commonly attributed to geopolymers (Cioffi et al., 2003; Duxson et al., 2006; Abdullah et al., 2012; Nyale et al., 2013; Singh et al., 2015). The raw materials, processing conditions and solution composition have an influence on the setting time of the geopolymer, as mentioned in section 2.5.1.

2.6. Effects of Geopolymer Processing Conditions

The synthesis of a geopolymer has three factors that substantially affect the geopolymerisation process. The three factors such as type and concentration of alkali metal ions, curing conditions and silicate concentration all depend on the materials used from the initial stage. The ratio of these to the CFA has a great impact on the properties of geopolymer synthesised. This section will briefly explain the effect of each variable on geopolymer synthesis.

2.6.1. Chemical Parameters

i. Effect of Sodium Hydroxide

In the aluminosilicate binder or CFA in this case, the sodium cation is responsible for the escalation of the dissolution phase because of its interaction with silicate and aluminate anion. The potassium cation is responsible for the increases of the gel network due to its connotation with larger aluminosilicate anions (McCormick and Bell, 1989). The aforementioned behaviour of metal cations is observed in geopolymerisation. Van Jaarsveld and Van Deventer (1999) studied the behaviour of Na^+ and K^+ during the dissolution stage and observed that with Na^+ the dissolution of aluminium and silicon precursors is larger than with K^+ .

The concentration of NaOH is one of the most significant factors for the geopolymerisation formation process. The NaOH concentration increases the solubility of the CFA. The effectiveness of the geopolymerisation is promoted by NaOH concentration content in the process, therefore, the increase in NaOH concentration directly increases the dissolution rate of silica and alumina ions from CFA. The initiation of oligomers is critical and is promoted by

an increase in Si and Al contents in the aqueous phase. This step is essential as it leads to the polycondensation step which is for strength development of the geopolymer (Yahya et al., 2014; Panias et al., 2007). Concluding on abovementioned analysis, it can be said that the increase of NaOH concentration in the aqueous phase can have a direct effect on mechanical properties of the geopolymers.

According to results of a study conducted by Panias et al. (2007), there is a decrease in compressive strength as the concentration continues to increase beyond an optimum. As discussed above, NaOH is the initiator of the dissolution stage which releases the Si and Al species in the solution and forms oligomers; these will lead to polycondensation, although NaOH promotes this process if too much NaOH concentration inhibits this process. In extremely high alkaline conditions, oligomeric silicate species become unstable in favour of mononuclear silicate species. As a result, in high alkaline conditions the equilibrium shifts towards mononuclear species, reducing the oligomers of silicate species in aqueous phase and thus decelerating the effectiveness of the polycondensation process, which is measured by the compressive strength of the geopolymeric materials (Hamidi et al., 2016; Rattanasak & Chindapasirt, 2009).

ii. Effect of Alkali Silicate Concentration

When the concentration of silicate is increased, the physical properties of the CFA-based geopolymer have been improved significantly (Hamidi et al., 2016; Rattanasak & Chindapasirt., 2009). Fansuri et al. (2010) studied the effect of silicate concentration in geopolymerisation and deduced that optimal geopolymerisation is highly dependent upon the homogeneously dispersed alkali (MOH, where M = Na, K) in the silicate concentration. Soluble silicate is a vital factor in the geopolymerisation process. Soluble silicate specie is responsible for initiating the oligomerisation formation stage. The oligomerisation stage is responsible for forming monomers, dimmers, trimmer, and tetramers, enhancing the geopolymerisation efficiency. The addition of soluble silicate to the geopolymerisation process was first studied by Rahier et al. (1996, 1997). The compressive strength of geopolymers increased almost linearly with the increase of Si in the aqueous phase even though incessant increase in silicate content diminishes the mechanical strength of the geopolymers.

Hamidi et al. (2016) stated that an intensification of SiO_2 to Na_2O concentration ratio results in a chemical shift from the monosilicate chains to cyclic trimers species caused by the addition

of soluble sodium silicates in the geopolymeric system, increasing the mechanical properties of the resulting geopolymeric materials. When NaOH concentration is kept constant in the system the continuous addition of soluble sodium silicate results in the positive effect of the mechanical properties of the geopolymers. Although increasingly adding soluble sodium silicate influences the geopolymer mechanical strength positively, there is an upper limit with the reaction which is determined by the viscosity of the geopolymer paste. The continual increase of sodium silicate increases the viscosity of the geopolymer paste, resulting in reduced workability of the paste. This causes difficulty in moulding, and it will weaken the geopolymer's mechanical properties (Rattanasak & Chindaprasirt., 2009).

Geopolymerisation is a process that occurs when an alkali aluminosilicate source is mixed with alkali activator (NaOH/KOH). The 1st stage of geopolymerisation is the dissolution stage; hydroxide ions are responsible for breaking down the alumina and silica bonds from the aluminosilicate source (CFA). It is necessary to keep the SiO₂ to Na₂O ratio to allow the polycondensation stage to escalate, therefore the need to add soluble sodium silicate is important. The high concentration of NaOH can negatively influence the geopolymerisation process due to more hydroxide ions that will hinder the reaction from occurring and decreasing the strength of the geopolymer.

Kaur et al. (2018) studied the effect of NaOH and Na₂SiO₃ on the mechanical properties of CFA-based geopolymer. The effect of NaOH on the compressive strength was studied and as the concentration of the NaOH increased, so did the compressive strength. The authors concluded that the accumulation of sodium silicate improved the strength development in geopolymer mortar. As the Na₂SiO₃/NaOH ratio increased to 3,5 there was a notable change rather decrease in compressive strength; this can be a result of high alkali content, which hinders/stops the geopolymerisation process (Vora & Dave, 2013; Patankar et al., 2014; Abdul Rahim et al., 2014; Degirmenci, 2017).

iii. Effect of Water Content

Many studies have shown that as the water to binder ratio increases, the compressive strength decreases. This observation is attributed to the effect water has on the NaOH concentration, which increases substantially when less water is added to the process. Furthermore, an increase in the water content in the geopolymerisation process results in decreases in the initial silica concentration in the aqueous phase, which negatively affects the process of oligomer

formation, polymerisation and geopolymer setting time. In conclusion, the reduction of the water in the system positively influences the oligomers to polycondensation and the setting time of the geopolymer paste.

Panias et al. (2007) proved that mechanical strength improved as the water content decreased. The continuous decrease in water content affects the system negatively as this will minimise the complete mixing of CFA with alkali silicate, causing insufficient strength of the geopolymer. Insufficient water reduces workability, making the molding process difficult and resulting in voids and gaps in the geopolymer, which negatively affects mechanical strength. The water content of the geopolymerisation process has to be sufficient to improve the workability of the paste but not too much to weaken the mechanical strength of the geopolymer. The water content can also be affected by the compacting method used. If pressing is used, less water can be used but if the tamping method is used, a little more water has to be added.

2.6.2. Physical Parameters

i. Effect of Curing Temperature

High temperature curing increases the rate of geopolymerisation, producing adequate strength (Rowles & O'Connor, 2003). Patankar et al. (2014) studied the effect of curing temperature on compressive strength. The compressive strength increased as the curing temperature increased. The curing process is vital for controlling the content of water during the polymerisation step, during which the geopolymer hardens. Water has a great influence upon alkaline activation reactions therefore humidity of the geopolymer has an effect on the structure and the mechanical properties of the geopolymers. Various authors have studied the effect that temperature has on the structure and mechanical properties of CFA-based geopolymer products (Muñiz-Villarreal et al., 2011; Vora & Dave, 2013; Mo et al., 2014).

2.7. Geopolymer Applications

Geopolymers have a range of properties and characteristics such as low cost and low energy consumption during synthesis that makes the geopolymers most appropriate for various applications, depending on the raw materials used in polymer synthesis (De Silva et al., 2007). Since the discovery of geopolymers by Joseph Davidovits many geopolymerisation reactions have been used to produce different types of geopolymers for different applications such as concrete and cement, resin and binders and art and archaeology. Geopolymers can be pure, with fillers, or reinforced, depending on their application. Raw materials used for geopolymerisation will determine the final products. The Si:Al ratio governs the application of the geopolymer product. Table 2.6-1 shows different Si/Al and geopolymer applications. The silica to alumina ratio has a huge impact on determining the final structure of the resultant geopolymer materials, and this also influences the application of geopolymers (He et al., 2012; Kriven et al., 2003; Duxson et al., 2005).

Table 2.7-1: Si: Al ratio and their applications. Adapted from Duxson et al. (2005).

Si: Al ratio	Applications
1	<ul style="list-style-type: none"> • Ceramics • Fire Retardant • Bricks
2	<ul style="list-style-type: none"> • Toxic Waste Encapsulation • Radioactivity • Low carbon dioxide emitting geopolymers
3	<ul style="list-style-type: none"> • Fire resistant fibre glass • Foundry equipment • Heat resistant composites, 200°C to 1000°C • Tooling for aeronautics titanium process
>3	<ul style="list-style-type: none"> • Aeronautics tooling
20-35	<ul style="list-style-type: none"> • Fibre composites with heat and fire resistance properties.

2.7.1. Arts and Archaeology

Geopolymer-based artifacts are reported to resemble natural stone. As a result, many artists have replicated their artwork in silicone-based rubber moulds. In the 1980s an artist named Georges Grimal did multiple stone formulations. Geopolymeric materials can be used for residential and art gallery decorations including archaeology and cultural decorative designs. Joseph Davidovits claimed that the Egyptians were aware of these properties and generated a geopolymeric reaction when producing the limestone blocks (Geopolymer Institute, 2018).

2.7.2. Geopolymer cements and concretes

The words geopolymer cement and geopolymer concrete are misunderstood as people often refer to them as the same thing. Concrete is a composite material that consists of cement, aggregates (sand, stones, glass, etc.) where cement is the binder. Geopolymer is a tridimensional alkali aluminosilicate binder that can be used as an alternate solution for use in construction and infrastructure (Dias & Thaumaturgo, 2005). Geopolymer relies on less processed industrial waste materials and can withstand some of the durability issues that conventional concrete currently faces. The geopolymerisation process requires aluminosilicate

sources such as coal fly ash, alkaline reagents such as NaOH, KOH and Na_2SiO_3 with a molar ratio $\text{SiO}_2: \text{M}_2\text{O} \geq 1.65$, M is Na or K) and water (Duxson et al., 2007; Turner & Collins, 2013).

Concrete is one of the world's most useful, robust and reliable building materials. After the transportation industry, the construction industry emits the second most carbon dioxide, contributing to the global warming crisis. With the positive outcome of geopolymer concrete, many possible applications are being investigated. The utilisation of CFA-based geopolymer in wall partitioning, furniture, insulating material, aerospace, and the automobile is a continuous investigation. Many different fibres have been added to geopolymer synthesis to increase durability, flexibility, tensile strength and to decrease the weight of geopolymer for application in indoor uses (Shaikh, 2013). The geopolymers with aggregates/additives are called fibre reinforced geopolymers. The aggregates range from steel fibres to waste paper fibres (Pan et al., 2011; Korniejenko et al., 2016).

2.8. Reinforced Geopolymer Composites

Concrete has low flexural and tensile strength, low strain capacity, and is brittle. Addition of natural or synthetic fibres improve the ductility of concrete with improved toughness, strain capacity and flexural strength. Reinforced geopolymers are significant in many industries e.g., in the aeronautical and automobile industries, naval architecture, and ground transportation (Shaikh, 2013; Natali et al., 2011). Geopolymer materials have advantages in comparison with traditional materials. The addition of fibres is an efficient method for improving such mechanical properties as fracture toughness (Bhutta et al., 2017; Korniejenko et al., 2016). The presence of fibres reduces the general effect of cracking, suppresses all brittle behaviour and enhances ductility (Shaikh, 2013; Ohno & Li, 2014). A range of fibres has been investigated for utilisation in concrete reinforcement over the years. The use of fibres for the improvement of mechanical properties in concrete has been proven to enhance mechanical properties and since then researchers have been investigating fibre reinforcement in geopolymer composites/alkali-activated composites.

2.8.1. Reinforcement Material Utilised in Geopolymer Development

Improving the mechanical properties of geopolymers has been a focus of research over the years. The idea of using fibres as reinforcing material in concretes to improve mechanical

properties started in the 1960s. The traditional fibres used are glass, steel, and carbon steel. The reinforced concrete has improved mechanical properties such as flexibility, impact resistance and abrasion.

Zhao et al. (2007) studied stainless steel meshes as reinforcement material for potassium silicate activated aluminosilicate binder (geopolymer). The specimens were cured at 80°C for 48 hours and processed with a vacuum bagging technique. The stainless steel fibre improved the ductility of the geopolymer at 1% stainless steel mesh, and the tensile strength of the samples improved even after the sample was exposed to high temperatures up to 1050°C. Bernal et al. (2010) studied the performance of alkali activated stainless steel fibre reinforced concrete compared to ordinary Portland cement and concluded that splitting, tensile and compressive strength improves with the incorporation of steel fibres at 28 days of aging. The concrete's flexural behaviour and water absorption improved remarkably with the slight addition of steel fibres. Due to the cost incurred by the rehabilitation and replacement of damaged stainless steel-reinforced concretes by corrosion, many engineers and researchers have sought viable alternatives such as substituting stainless steel bars with fibre reinforced polymer (FRP) bars.

Carbon has been investigated as a reinforcement material mainly in situations where fire-resistance is of concern. Lyon et al. (1997, 1996) conducted intense research on potassium aluminosilicate geopolymers to find their appropriate application in airplane cabins. When reinforced with carbon fabrics, the carbon reinforced geopolymer composite improved the flexural strength by 63% and no smoke emission, flame ignition and any burns were noticed when the material was exposed to stimulated fires. During this study other materials were investigated using carbon fibre as reinforcement material. These materials were tested for flammability and as a result, they caught fire and smoke was detected within 15 to 20 minutes of fire exposure. This proves that these materials will prevent passengers from escaping in cases where an automobile starts burning. On the other hand, geopolymer composites with carbon reinforcement posed no such threat.

Lin et al. (2008) did a study with a potassium silicate activated metakaolin geopolymer reinforced by carbon fibre sheets. The vacuum bagging technique was used to prevent carbon fibres fracturing when introducing a geopolymer paste in a mixer. The results showed that the tensile strength was improved but the fibre pull-out test was failed by the specimen. Lin et al.

(2010) further studied the infiltration of the geopolymer composite using a silicate solution. This treatment effectively filled the voids, increasing the density, compressive strength and flexural strength by 93%, 35% and 20% respectively.

Yeol Choi et al. (2005) investigated the utilisation of glass fibre as reinforcement material for geopolymer development. The investigation was mainly focused on the effect of glass fibre reinforced geopolymer to splitting tensile strength and compressive strength. The results demonstrated that tensile strength increased with the addition of glass fibre by 50% and compressive strength by 15%. Jagannadha Rao et al, (2010) studied the use of glass fibre as reinforcement material in recycled aggregate concrete. The content of glass fibre increased the compressive, flexural and tensile strength of the recycled aggregate concrete. The optimum values of all these mechanical properties were obtained at 0.03% of the fibre composition of the concrete.

2.8.2. Natural Fibre-Reinforcement Material in Geopolymer Development

Increasing environmental awareness and sustainability of construction materials has led to a considerable amount of research on alternative fibres to reinforce cementitious materials. Traditional fibres such as steel, carbon, and glass are slowly being replaced with natural fibres because they are abundant, reproducible, and environmentally friendly. Natural fibres have many advantages such as low density, high specific strength, low cost, renewability, and they reduce CO₂ emissions. Research has been done on different natural fibres such as coconut, sisal, hemp, cotton stalk, sorghum and vegetable fibres. Minimal study has been done on the utilisation of natural fibres to reinforce geopolymer.

Chen et al. (2013) investigated the use of sweet sorghum fibres as reinforcement to improve the mechanical behaviour of fly ash-based geopolymer. The results showed the weight of the geopolymer decreased with an increased sorghum content, whereas tensile and flexural strength increased only up to 2 wt% addition of sorghum fibres. Alomayri et al. (2013) studied the mechanical, physical and fracture behaviour of CFA-based geopolymer reinforced with cotton fibres from 0.3 wt% to 1 wt%. Results showed that the flexural strength and fracture toughness increased while the density of the geopolymer decreased as the fibre content increased. The optimum fibre content of the geopolymer was 0.5 wt%.

Alomayri et al. (2014) further investigated and improved the mechanical properties of cotton fibre- reinforced geopolymer. The results showed that all the mechanical properties of the composites were improved by increasing the cotton fibre contents. It was found that the mechanical properties of cotton fabric reinforced geopolymer composites were superior to the pure geopolymer matrix. Teixeira-Pinto et al. (2008) investigated the utilisation of jute as a reinforcement fibre for metakaolin-based geopolymeric material. Based on the results that were obtained, jute fabric can be used to enhance the mechanical properties of composite geopolymer and aid with fire resistance.

Reis (2006) investigated the mechanical properties of three different natural fibres such as banana, coconut, and sugar cane fibres. Shredded coconut fibre and sugar bagasse fibre increased the rupture properties, including modulus of rupture. Banana pseudostem fibre was used as reinforcement material and the fracture toughness did not increase. Coconut fibre reinforcement displayed a small increase in flexural strength compared to other synthetic fibres such as carbon and glass. Sugar cane bagasse and banana pseudostem fibre resulted in weakening the flexural strength of the composite geopolymer.

On the other hand, the durability of natural fibre as a reinforcement material is of concern. The lignin and hemicellulose could dissolve if in contact with the alkaline pore water in the geopolymer or cementitious material and this could result in a weaker material. A poor matrix is formed by poor interface quality between fibre and cement. The compatibility of the interfaces of the materials also plays a role in the performance of the composite. In addition to concerns about durability, most of these fibres are from plants, fruits, and vegetables which rely on seasonal changes which result in fibre shortages in some seasons. In view of the abovementioned shortcomings of the use of natural fibres as reinforcement material, considerable research has been conducted to look for alternative raw materials that are cost-effective, available and durable. Waste materials such as paper and plastics from cardboard boxes and plastic bottles have been investigated for their use in the construction industry as aggregates and composite material.

2.8.3. Waste Material as Reinforcement Material in Geopolymer Development

Large amounts of mass solid waste such as paper, plastic, concrete, metal, and glass are continuously being produced around the world and in South Africa due to continuous population growth and urbanisation (Safiuddin, et al., 2010). As the literature review shows, the aforementioned waste materials can be used in the construction industry to enhance engineering properties. This method results in waste management as most of the worst materials will be utilised rather than distributed to infills (Okeyinka, 2016). Various waste materials can be utilised in the construction industry, therefore the possible utilisation of solid waste in this sector stands to be a viable option for its disposal.

Table 2.8-1: Solid waste materials with their applications in the construction industry

Mass Solid Waste	Recycling Method	Application in Building Material	References
Plastic	- Crushed	A resin binder	Sam & Tam., (2002)
	- Aggregate	Fine aggregate	Kamaruddin et al. (2017)
Metal	- Grind to powder	thermoformable	
	- Melt	Blended cement,	Mo et al. (2016)
	- Reuse	recycled steel, lightweight concrete	Bernal et al. (2010)
Glass	- Crushed aggregate	Tile, paving block	Alomayri, (2017) Shaikh, (2014)
	- Reuse		
	- Grind to powder		
Paper	- Paper pulp	Wall panel, building blocks, composite panels, and fibre reinforced composites, Fibre reinforced composite	Pachamutu & Thangaraju. (2017)
	- Paper fibre		
	- Paper ash		Kakali et al. (2018)

2.9. Waste Paper in Construction

The general definition that best describes waste paper is paper that cannot be used for the purpose intended. It is generated mostly by human activities, and as the population continues

to grow, waste paper production is also increasing. It has drawn interest in the construction industry as a component of composite materials. Research has found that paperboard composites are ideal for commercial building, household, electrical and packaging applications due to their quality, ease of processing and low cost of production compared to traditional materials (Pervaiz and Sain, 2003). Literature demonstrates that waste paper can be used as a building material, as pioneers have created different types of boards over the years. Waste paper has been used with cement mainly to make papercrete which is a composite of paper and cement. Researchers have made paper boards with better mechanical properties, and low thermal conductivity (Okeyinka & Idowu., 2014; Sangrustamee et al., 2012; Shafiur Rahman et al., 2014). These paper boards are mainly used for indoor purposes such as furniture, ceiling, and wall partitioning. Waste paper materials or furniture are susceptible to fire, therefore measures should be in place to avoid the spread of fire in cases of emergency. This research focuses on waste paper as an aggregate to coal fly ash for the development of lightweight geopolymer.

2.9.1. The Availability of Waste Paper

In most developed and developing countries, waste paper-based goods constitute a large percentage of municipal solid waste (MSW). Globally waste paper is the second largest component of total waste composition and it is estimated that it will continue to increase in both developed and developing countries as paper products are in high demand due to their desirable strength to weight ratio and cost-effectiveness (Tisserant et al., 2017).

In South Africa, waste paper has become one of the largest components of the municipal solid waste stream. Waste paper and paper board generation from 1960 until 2010 has moved from 20% to 30% respectively. In 2011 about 21% of the MSW consisted of waste paper and paper board and was the second largest component in MSW. Currently waste paper accounts for about 42% of litter in streets (Burnely et al., 2007).

About 2.2 to 2.4 million tonnes of paper were consumed in South Africa in 2014. 72% of the paper produced is recyclable and can be used for various applications (PRASA 2015). 430 000 tonnes are available for recycling in Western Cape province (Godfrey & Oelofse, 2017). Waste paper is currently being considered as an alternative for fibre by many paper manufacturers. 65% of South African mills are using it for manufacturing corrugated boxes, coffee cup holders,

books, cereal boxes, and soap cartons. There is a noticeable growth in tissue paper, and its potential use in the treatment of waste is being investigated, provided that this can be properly and economically separated (e.g. collected with an anaerobic fraction for anaerobic digestion, separated at source or managed with residual waste) (Godfrey & Oelofse, 2017; Larney & van Aardt, 2009; Nahman & Godfrey, 2010; Basson et al., 2015).

2.9.2. Environmental Impact of Waste Paper Disposal

Landfill space for waste paper disposal is limited as paper production and consumption is increasing daily as discussed above. Therefore, it is important to explore alternatives to recycling waste paper for productive use. It was estimated that by 2020, paper production companies will be producing 500 million tons papers per year. There is a daily increase in demand for this product and reduction or recycling needs to be planned (Basson et al., 2015). The main environmental problems caused by paper waste are land and water pollution. Paper and pulp are considered the third largest forms of industrial pollution of water, air and soil. Bleaches based on chlorine are used during the production process, resulting in toxic materials that have a negative impact on the environment. When paper rots, it produces 25 times more toxic methane gas than carbon dioxide (Levis & Barlaz, 2011).

At present, the ocean is filled with plastic and paper waste (about 8 million metric tons per year) which is interfering with aquatic life. It is predicted that at the rate at which water pollution is increasing, by 2030 there will be more paper and plastic than aquatic life (Nahman & Godfrey, 2010; Schyns et al., 2017). To resolve rising environmental problems caused by the disposal of waste materials from both the paper and the construction industry, it is paramount to recycle waste paper (WP) in the construction industry. This will not only solve the disposal problem of paper but it will improve the engineering properties of construction materials whilst decreasing the negative impact that the construction industry has on greenhouse gas emission, with high natural resource and energy consumption (Pillay, 2018).

2.9.3. Waste Paper in Geopolymers

According to research, waste paper can be used in the construction industry to make various types of building materials due to its desirable properties for utilisation in the construction industry (Yun et al., 2007; Fuller et al., 2006; Decard et al., 2001). The continuous increase of

waste disposal problems can be addressed in a sustainable way through the application of waste paper in the construction industry to make many products such as wall board, furniture and ceilings. Okeyinka (2016) confirmed that a combination of waste paper sludge ash (WSA) can be combined with other waste materials to produce a concrete binder.

Nidzam & Kinuthia et al. (2010) and Abdullah et al. (2013) report that an eco-friendly binder was produced using GGBS and WSA. The binder produced possesses better engineering properties than the conventional Portland cement-based concrete, including its appearance, strength, and durability. Geopolymer has been synthesised by a few researchers (Kakali et al., 2018; Yan & Sagoe-Crentsil, 2012) using coal fly ash and different lightweight aggregates for the purpose of developing a construction material that can be utilised for lightweight applications including wall partitioning. The use of coal fly ash and waste paper in the building sector is beneficial for the environment as both coal combustion by-product and waste paper are being utilised therefore preventing the emission of pollutants to the water, soil and air. The construction industry will minimise its use of raw materials and this will produce a profit for the industry. Waste paper properties such as low density, desirable strength-to-weight ratio, insulation properties, low cost, and availability are the primary advantages of utilising WP in the construction industry.

WP in the construction industry has its advantages but it also has disadvantages in certain applications. One of its main disadvantages of using waste paper based geopolymers or construction materials as furniture is the high moisture absorption which will weaken the structure to the point of failure. This drawback can be attributed to the nature of paper which is a high water and moisture absorption material. The paper fibre absorbs more water than cement or geopolymer uses for hydration and this results in reduction of mechanical strength properties. Many of the manufactured composites have a very low percentage of paper and a high percentage of coal fly ash and sand (Kakali et al., 2018; Yan & Sagoe-Crentsil, 2012). Despite the negative properties of waste paper as a construction material, research has shown that waste paper possesses several attractive properties which allows it to be used for lightweight wall partitioning, ceilings, thermal insulation, low cost housing, and sound absorption. Although waste paper can be used to produce many construction materials, this study focused on finding the optimum conditions for developing a lightweight geopolymer using waste paper and CFA.

2.10. Properties of Geopolymer

Geopolymer applications are mainly for the construction industry therefore it is of paramount importance to enhance the mechanical properties of geopolymers like cement and concrete. Various studies have reported that fly ash based geopolymers possess better properties than Portland cement including high compressive strength, low shrinkage, and low water absorption.

2.10.1. Compressive Strength

The compressive strength of a CFA-based aluminosilicate binder depends on many factors such as curing temperature, alkali concentration, curing time and various aggregates and additives. Alkali concentration type influences the amount of Si and Al to be released during the geopolymerisation process (de Vargas et al., 2011). Na_2SiO_3 is combined with a sodium hydroxide solution to help with compressive strength due to its ability to increase the solution's viscosity, enhancing gel formation during the geopolymerization process. As depicted by Momen et al. (2013) in figure 2.9-1 and Livi & Repette. (2017) in figure 2.10-2, the compressive strength of the geopolymer increased with an increase in NaOH concentration. This is due to the ability of hydroxide ions to enhance the dissolution stage during geopolymerisation. As the concentration of NaOH continues to increase there are more hydroxide ions which will hinder the dissolution stage from occurring, causing an ineffective reaction resulting in low compressive strength. Figures 2.10-1 and 2.10-2 show a typical trend of the effect of sodium hydroxide concentration on compressive strength.

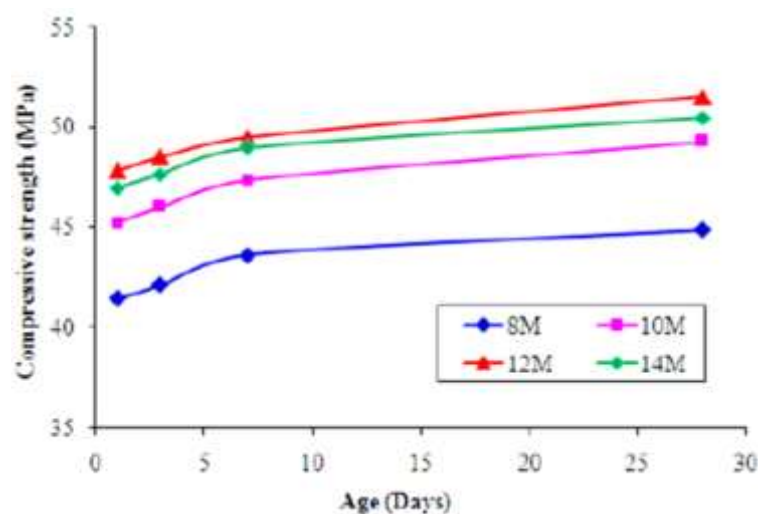


Figure 2.10-1: The effect of NaOH on Compressive strength. Adapted from Momen et al. (2013).

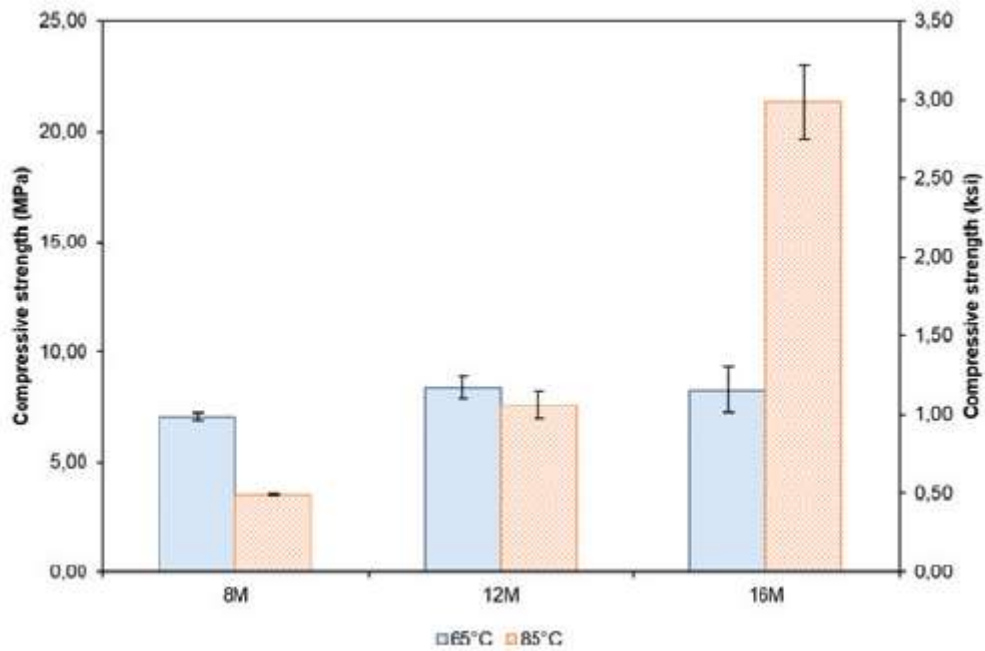


Figure 2.10-2: The effect on NaOH on Compressive strength. Adapted from Livi & Repette (2017).

Geopolymer compressive strength depends on the quality and quantity of the fibre reinforcement. Waste paper reinforced geopolymers have been studied for a decade now and it is known that the compressive strength is also dependent on waste paper content. Figures 2.10-3 and 2.10-4, adapted from Srichandr et al. (2012) and Thangaraju & Pachumut (2017) respectively, shows that typically as the content of waste paper increased the compressive strength decreases. Figures 2.10-3 and 2.10-4 show a typical trend of the effect of waste paper content on compressive strength.

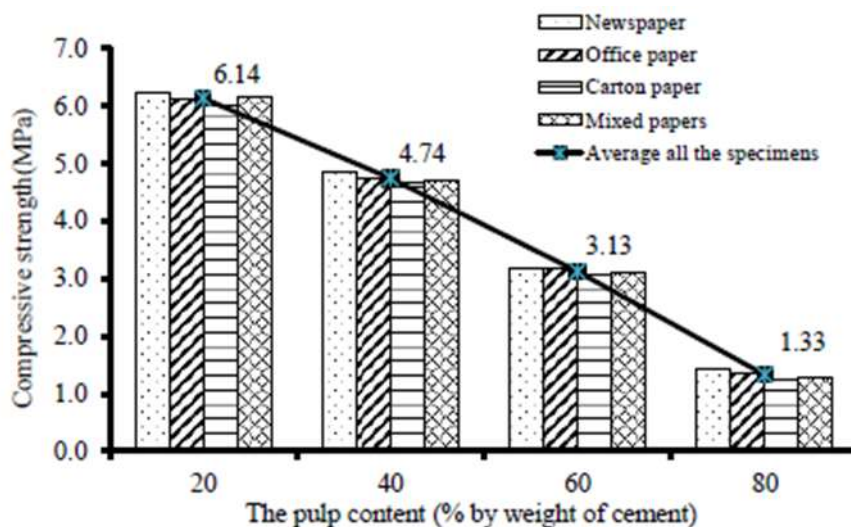


Figure 2.10-3: Effect of Waste paper content on compressive strength. Adapted from Srichandr et al. (2012).

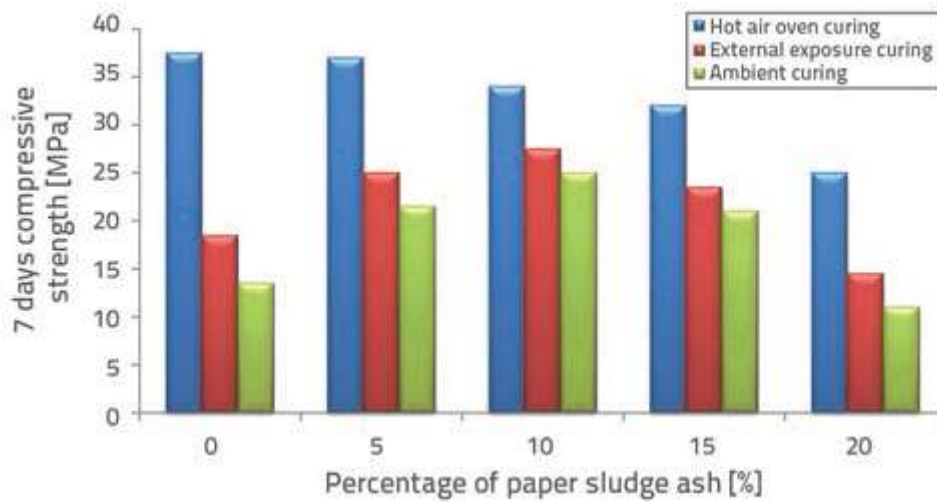


Figure 2.10-4: Effect of water paper content on compressive strength. Adapted from Thangaraju & Pachumut (2017).

Curing temperature also affects CFA-based geopolymer compressive strength. At high curing temperatures, geopolymer strength is also high. This is due to the collapse of the capillary pores resulting in a dense structure (Leung and Pheerapha, 1995). Geopolymers can be healed at room temperature, but the geopolymerization reaction is slow, requiring healing time (Somna et al., 2011). As the temperature increases, the granular structure is broken down by dehydration and excessive shrinkage, and the compressive strength is finally reduced, as demonstrated by Bakria et al. (2011) in Figure 2.10.5. Figure 2.10-5 shows the typical pattern of the effect of curing temperature on compressive strength.

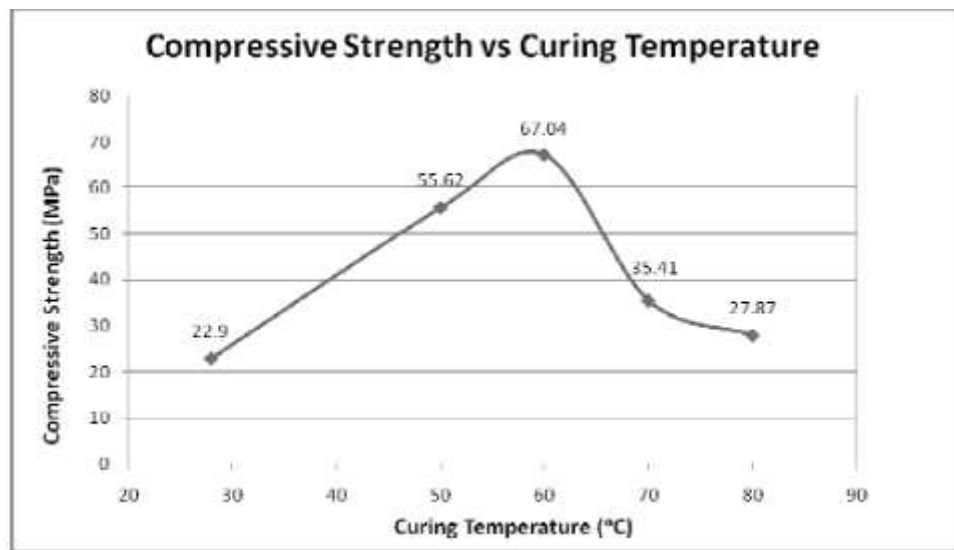


Figure 2.10-5: Effect of temperature on compressive strength. Adapted from Bakria et al. (2011).

2.10.2. Shrinkage

As the geopolymer and concrete dry, the concrete decreases in volume which is called shrinkage. There are different types of shrinkage that concrete can experience such as thermal, chemical, plastic and drying shrinkage (Gilbert, 2002). Drying shrinkage occurs when the reduction in volume is observed due to the loss of water during the drying process. Among all the shrinkage types, drying shrinkage is accountable for the total long term shrinkage. Many factors affect drying shrinkage, mainly water content and aggregate type. Aggregates play a major role in impacting concrete shrinkage (de Larrard et. al., 1994; Neville, 2000). High aggregate shrinkage results in smaller shrinkage, because of aggregate materials of high modulus and rougher surfaces. The higher water to concrete proportion typically brings about higher shrinkage because of interrelated impacts. As water to binder proportion expands, paste quality and firmness reduce and as the water content builds, shrinkage potential expands in light of the fact that it additionally decreases the volume of limiting totals (de Larrard et. al., 1994).

2.10.3. Water Absorption

The absorption of water depends on the pore structure of the material. Olivia et al. (2008) examined the capillary water absorption of a fly ash geopolymer concrete comprising low calcium. The conclusion drawn is that CFA geopolymer concrete exhibits low water absorption and sorptivity. For the capillary porosity and the total porosity of geopolymer concrete, low water-binder ratio and better grading are recommended. Figure 2.10-6 below depicts a typical trend of the effect of water absorption on the samples of different paper types and content. The results show that as waste paper content increases, water absorption increases. Although there are small variations in specimen properties for different waste papers, the cardboard-based paper seems to have high water absorption compared to the rest.

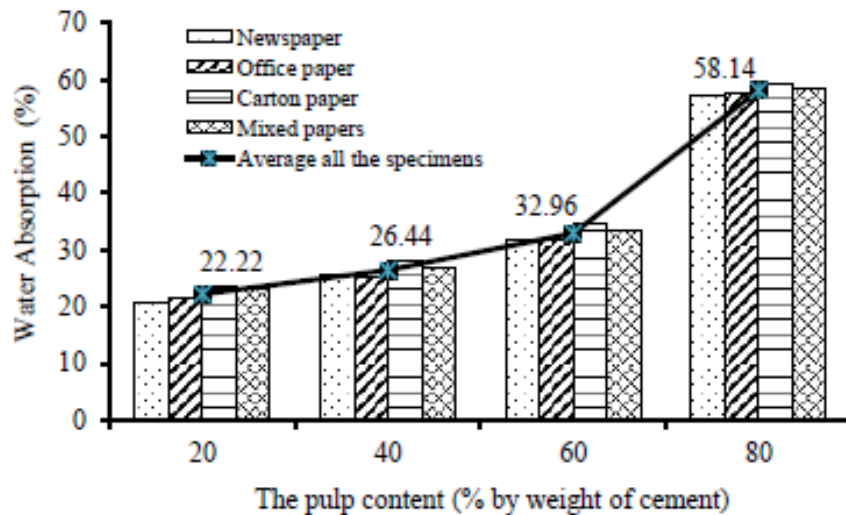


Figure 2.10-6: Effect of waste paper content on water absorption. Adapted from Srichandr et al. (2012).

2.11. The Current State of the Art with Respect to the Geopolymer-Paper Waste Composite

Waste paper is used in the construction industry by making what is known as papercrete for various uses such as the development of ceilings, lightweight partition walls, infilled walls and basements in non-earthquake prone areas. Fly ash has characteristics that are similar to ordinary Portland cement and literature shows (Mackenzie & Welter, 2014; Nyale et al., 2013; Duxson et al., 2006; Bamkwocki et al., 2004) extensive theoretical background that has been developed on fly ash replacing ordinary cement. This research is focusing on using the above-mentioned two waste materials to develop a low-cost lightweight geopolymer rather than disposing the wastes into the environment.

Waste paper-based construction materials have been developed over the years for different purposes in the construction industry. Papercrete has been widely used for non-load bearing construction material and the composition has included ordinary Portland cement and other aggregates. Various studies have been conducted on waste paper as a construction material to develop papercrete using cementitious material, for instance, coal fly ash, or bottom ash. These studies inform the current project.

Gailius & Laurikietytė (2003) studied the compressive strength and workability of concrete binder synthesised using WSA and GGBS respectively, at two waste paper to blast furnace slag ash ratios of 0,5 and 0,4. The results show that after 28 days of aging, compressive strength was in the range of 15 and 21 MPa at 0.4 ratios of waste paper to slag content. Akhtar et al.

(2011) investigated the effect of waste paper pulp on the weight of the fly ash based geopolymer and found that the weight of the brick was reduced by 50% compared to the reference brick. Yan et al. (2012) used dry waste paper sludge to prepare a lightweight geopolymer. Because waste paper absorbs water, the continuous increase of waste paper sludge in the paste reduced workability. The average compressive strength and weight of mortar samples incorporating 10 wt% waste paper sludge were 25 MPa and 5% less than the reference weight.

Yan et al. (2012) used dry waste paper sludge for lightweight geopolymers. Waste paper added to the mortar mix reduced the geopolymer mortar workability. For 25% and 10% waste paper content the compressive strength was 92% and 52% of the reference geopolymer strength after a 91-day aging period. The drying shrinkage percentage of the geopolymer decreased by 34% for 10% waste paper content and 64% for 25% waste paper. Based on the results, excluding the high moisture absorption, waste paper appeared to be compatible with geopolymer chemistry which proves waste paper to be a good naturally based aggregate for geopolymers.

Antunes Boca Santa et al. (2013) studied the effect of paper sludge on the geopolymer mechanical properties. Bottom ash was used for the aluminosilicate source, sodium hydroxide ($\text{NaOH} = 5, 10 \text{ and } 15 \text{ M}$) and sodium silicate ($\text{Na}_2\text{SiO}_3, \text{SiO}_2/\text{Na}_2\text{O} = 1.58$) were used as alkali silicate medium. For the reactivity of the partially crystallised bottom powder, calcined paper sludge was used. XRF and XRD characterized solid waste and characterized the geopolymer specimens. 15 M NaOH and ratio of 2:1 of bottom ash and paper sludge were the best results obtained.

Anuara (2015) studied the effect of incinerated waste paper on geopolymer strength. Waste paper sludge ash and a combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were used as binders. Three different molarities of NaOH (8 M, 12 M, and 14 M) and different percentages of waste paper (0 %, 25 %, 50 %, 75 %, and 100 %) were tested. Experimental results showed the highest compressive strength of 6.788 MPa was obtained with the use of 12M NaOH. Momin & Sayyad (2017) investigated papercrete brick and the optimised mix was 1:1.5:4: 2 (cement: fly ash: sand: paper) resulting in compressive strength of 3.24 MPa which is higher than conventional clay bricks and satisfies the IS code recommendation. The development of fly ash and incinerated paper sludge ash mixture as source material for

geopolymer concrete preparation was investigated by Pachamutu & Thangaraju (2017). The samples are prepared under various curing regimes with a varying percentage (0 % -20 %) of fly ash replaced by paper sludge. The peak power was 20 MPa at 20%.

Table 2.11-1 shows studies done on waste paper reinforced geopolymers from 2003 till 2017. Almost all the researchers used incinerated paper before mixing with the aluminosilicate binder. Yan et al (2012) and Momin & Sayaan (2017) did not incinerate paper but used additives such as sand and cement and achieved 23 MPa and 3.2 MPa compressive strength.

Table 2.11-1: Literature Comparative Analysis from 2003 to 2017

Composite Structure (%w/w)				Alkaline Activator	Curing Temp (°C)	Curing time (hrs)	Aging Time (Days)	Compressive Strength (MPa)	Reference
Binder	Reinforcement	Water/Binder	Additives						
GGBS	Waste Paper Ash - 50%	0.5	Sand	-	25	-	7	19.3	Gailius & Laurikietytė (2003)
Fly Ash	Waste Paper- 10%	0.24	Sand	NaOH 12M & Na ₂ SiO ₃	60	24	28	25	Yan et al. (2012)
Bottom Ash	Calcined Paper- 33%	-	-	NaOH 15M & Na ₂ SiO ₃	60	24	7	15	Santa et al. (2013)
WSA	-	0.15	Sand	NaOH 12M & Na ₂ SiO ₃	70	24	7	7	Ridzuan et al. (2014)
Fly Ash	Waste Paper Ash- 50%	-	-	NaOH 12M & Na ₂ SiO ₃	60	24	7	7	Anuara (2015)
Fly Ash	Waste paper- 40%	-	Cement	-	25	-	7	3.24	Momin & Sayyad. (2017)
Fly Ash	Waste Paper Ash- 15	15%	Aggregates	NaOH & Na ₂ SiO ₃	60	24	7	20	Pachamutu & Thangaraju (2017)

2.12. Summary of Chapter

Based on the fibres used for reinforcement, geopolymers can be used for load-bearing materials such as walls and non-load-bearing materials such as wall panels, ceilings, etc. Fibres improve concrete ductility with improved tensile and flexural strength, load capacity, durability, and energy absorption. Reinforced alkali-activated materials/geopolymers play a significant role in many industries e.g., in the aerospace and automotive industries, naval architecture, and ground transportation (Shaikh, 2013; Natali et al., 2011). There is a possible use of waste paper to produce different types of building materials based on literature facts. Building materials based on waste paper have been confirmed to exhibit some desirable behaviors and properties for use in construction (Yun et al., 2007; Fuller et al., 2006; Decard et al., 2001).

The use of waste paper in the manufacture of building materials and other items can be viewed as a safe way to address the ever-increasing supply of waste paper worldwide, as long as the less suitable properties are strengthened. Table 2.11-1 shows the studies done on waste paper reinforced geopolymers from 2003 till 2017. Almost all the researchers used incinerated paper before mixing with the aluminosilicate binder. Only Yan et al. (2014) and Momin & Sayyad, (2017) did not incinerate paper but used additives such as sand and cement and achieved 23 MPa and 3.2 MPa compressive strength.

Literature has very limited information on the development of lightweight geopolymeric materials (LWGM) using waste paper and coal fly ash without additives or incinerating the paper. Published articles on waste paper as building materials are focused on using waste paper as ash/incinerated paper. The process of incinerating paper uses energy and this research looks at cost-effective methods of producing cheap but high quality material. Waste paper has been used in the building industry for the development of papercrete with cement being used as the binder (Fuller et al., 2006; Santamaria et al., 2007). Papercrete is a construction material made from a mixture of recycled paper, Portland cement, sand and other optional materials such as fly ash and styrofoam glass, etc. It is rendered by the inclusion of a recycled paper in the constituents of papercrete and its normal use as building blocks (Fuller et al., 2006) makes it of close relevance to the lightweight geopolymeric material being developed in this study.

The gaps being filled by this study have been discussed in section 2.8 of the previous chapter. Because of a lack of clear data, this study used the experience gained from the preliminary laboratory experiments in combination with the limited relevant/applicable knowledge from geopolymer making (as defined in the literature review) to improve the mix proportioning process for LWGM. A number of factors have been optimized in order to develop the lightweight geopolymeric material technology. The emphasis was on creating an effective mix proportioning system for the block prior to the start of the key experiments scheduled to resolve the research goals outlined in Chapter 1.

The current study is focusing on reducing the use of chemicals and materials as one of the objectives is to develop a cost-effective product. The material being developed in this current study will consist only of CFA and unincinerated waste paper with NaOH and Na₂SiO₃ as the only chemicals. The aim is to develop a lightweight and cost-effective geopolymer. The addition of chemicals and aggregates will result in a more costly geopolymer. To achieve a low cost and lightweight geopolymer, waste paper and fly ash will be the main raw materials. With regard to the environment, it is advisable to use fewer chemicals. therefore this research will also consider reducing sodium silicate content in geopolymerisation. The literature on the use of CFA and waste paper for the synthesis of lightweight construction material is limited, therefore most of the formulations in this current study are entirely new although some parameters were based on literature.

Chapter 3: Research Methodology

3.1. Overview

The section explains the nature of the geopolymeric material production approach and the experimental work that was performed. Next, the components are clarified: the proportions of the mixture, the processing, and curing of the sample samples (sections 3.3, 3.4 and 3.5). A description of the types of samples used, test parameters and test procedures (sections 3.7 and 3.8) of the lightweight geopolymeric material being produced in this research is then followed.

3.2. Experimental Methodology

3.3. Materials and Equipment

This study utilises coal fly ash (CFA) as the binder, waste paper (pulp) as filler material, sodium hydroxide and sodium silicate as alkali activator for CFA; last but not least, water was used for preparing the pulp and to improve workability as the paper absorbed water. The materials used in this study include coal fly ash, waste paper, sodium hydroxide, sodium silicate, and water. The materials used in the manufacturing of lightweight geopolymeric material (LWGM) were based on the sourcing approach suggested by the United Nations General Assembly in 2015 for sustainable construction (Guterres & Zhenmin, 2019). As cement releases carbon dioxide, fly ash has been chosen to act as a binder and substitute for Portland cement. Coal fly ash was used as a cheap alternative because it is a waste product from power plants and has been documented to cause environmental pollution that puts people and marine life at risk. Coal fly ash is described in detail in Chapter 2. Waste cardboard was used to serve as the aggregate for the lightweight geopolymer due to its availability and lightweight properties (Fuller et al., 2006). Chapter 2 describes waste paper advantages in the construction industry in detail. Sodium hydroxide and sodium silicate were used for activating CFA. Water was used to improve the geopolymer paste's workability as a lot of water is consumed by waste paper.

3.3.1. Coal Fly Ash Sampling

The CFA used for all experiments is class F and it is from Lethabo Power Station, located close to the Vaal River near Vereeniging in the Free State province in South Africa. The fly ash samples were kept in sealed plastic containers away from sources of moisture and these containers were stored in a dark cool cupboard to protect them from any temperature variations.

3.3.2. Waste Paper Sampling

Post-consumer waste paper pulp (i.e. cardboard boxes) (Figure 3.2) collected from a recycling company in Montague Gardens, Cape Town, South Africa was used for the lightweight aggregate. Before its application in the laboratory experiment for the manufacture of LWGM, it was considered important to process waste paper in a usable form, since the cardboard was

obtained in the form of sheets. The cardboard boxes needed to be prepared into a pulp first prior to mixing. Due to a lack of proper equipment, Gayatri Papers provided the researcher with ready made pulp. The pulp is basically a mixture of paper and water. The pulp samples were kept in sealed containers to avoid moisture loss.

3.3.3. Chemicals

The list of chemicals and reagents used in this study is depicted in Table 3.3-1.

Table 3.3-1: Chemicals used in geopolymer synthesis

Chemical	Purity	Supplier	Location
Sodium Silicate (Na₂SiO₃) liquid	- Na ₂ O = 9.2 % - SiO ₂ = 30 % - H ₂ O = 60.8 %	Kimix	Cape Town, Western Cape, South Africa
Sodium Hydroxide (NaOH) pellets	98%	Kimix	Cape Town, Western Cape, South Africa
Shutter Oil (Releasing Agent)	-	Kimix	Cape Town, Western Cape, South Africa

3.3.4. Equipment

Table 3.3-2 shows the equipment used in the current study

Equipment	Supplier	Location
Imer Mix 60	Scientific Manufacturing	Cape Town, Western Cape, South Africa
Manual Hydraulic Press	-	Cape Town, Western Cape, South Africa
Hot Air Oven (EcoTherm)	Labotec	Cape Town, Western Cape, South Africa
Plastic and Nylon Moulds	Paktool Engineering	Cape Town, Western Cape, South Africa
Paint Brush	Kimix	Cape Town, Western Cape, South Africa
Plastic hand trowel	Kirk Marketing	Cape Town, Western Cape, South Africa
Beakers	Kimix	Cape Town, Western Cape, South Africa

3.4. Equipment Used

3.4.1. Hot Air Oven

A 24 litre Labote EcoTherm hot air oven used to dry geopolymeric material at the desired temperature.



Figure 3.4-1: EcoTherm drying oven: (A) main switch I, (B) control, (C) door.

3.4.2. Imer Mixer

Imer mix 60 was used for mixing all the solid materials and liquids to form a paste. The mixing unit consisted of one motor, one stirrer, and the plastic tank. The mixture made of coal fly ash, waste cardboard pulp, sodium hydroxide, sodium silicate and water were fed into the tank. The slurry solution was then stirred in the tank for 45 minutes in order to completely dissolve the raw material. This process is known as polycondensation or geopolymerisation; it results in a higher mass transfer rate and mixing intensity.

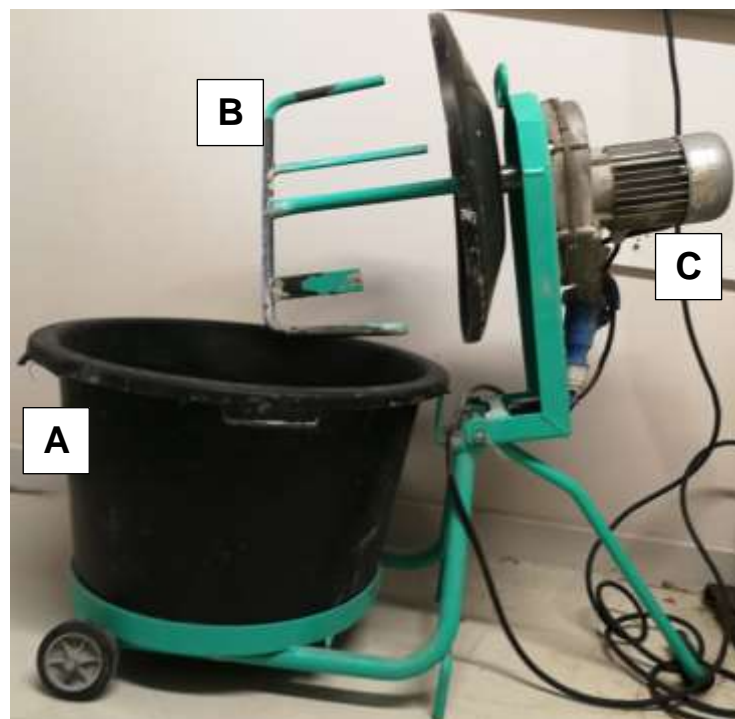


Figure 3.4-2: Mixing unit constitutes of (A) tank, (B) mechanical stirrer and (C) motor.

3.4.3. Other Equipment

Table 3.4-1: Other equipment used

Equipment/Item	Model	Details
Spatula	Flat and spoon spatulas, Lasec Co.	Stainless Steel
Stirring Rod	Glass rods, Lasec Co.	25 mm glass rod
Mixing Vessel	Beakers, Lasec Co.	2 litres

3.5. Laboratory Experimentation Procedures and Optimisation of Synthesis

Conditions

The research in the laboratory was to study the parameters that affect compressive strength, water absorption, weight, and combustibility. This section will show the optimisation of various formulations such as variations of parameters, synthesis curing conditions, and step by step procedures followed during the synthesis of the geopolymer. After the synthesis procedures are explained thoroughly the characterisation techniques used for analysing of raw materials and produced lightweight geopolymeric materials are described in this section. The properties tested include compressive strength, water absorption, weight, and reaction to fire.

In this investigation, CFA from Lethabo and waste-cardboard from Gayatri Papers were used as raw materials in the optimisation of the best formulations for the synthesis of lightweight geopolymeric materials. The lightweight geopolymeric material was synthesised by activating CFA with an alkali activator followed by adding waste-cardboard paper. To determine the best formulation for the production of LWGM, four parameters that were investigated such as the NaOH concentration, water content, curing temperature and waste paper content. The experiments were carried out by varying each parameter individually and keeping the other parameters constant.

3.5.1. Description of the Manufacturing Procedure

The liquid preparation is described in detail in the following sections, starting with NaOH preparation prior to mixing with Na_2SiO_3 . In this sample, concentrations of NaOH were 10 M, 12 M, and 14 M. Below are described the preparation of these concentrations.

3.5.1.1. NaOH Content

10 M of NaOH

To prepare a concentration of 10M of NaOH solution, 400 grams ($10 \times 40 = 400$ g) of NaOH pellets were mixed with one litre water; 40 is the molar mass of NaOH. The solution volume differed with the amount of FA to be activated. The amount of CFA varied based on the amount of waste cardboard paper percentage.

12 M of NaOH

To prepare a concentration of 12M of NaOH solution, 480 grams ($12 \times 40 = 480$ g) of NaOH pellets were mixed with one litre water; 40 is the molar mass of NaOH. The solution volume differed with the amount of FA to be activated. The amount of CFA varied based on the amount of waste cardboard paper percentage.

14 M of NaOH

To prepare a concentration of 14M of NaOH solution, 560 grams ($14 \times 40 = 560$ g) of NaOH pellets were mixed with one litre water; 40 is the molar mass of NaOH. The solution volume differed with the amount of FA to be activated. The amount of CFA varied based on the amount of waste cardboard paper percentage.

The NaOH solution was made 24 hours prior to geopolymer synthesis to allow the mixture to cool off as the reaction of NaOH and H_2O is exothermic. The Na_2SiO_3 to NaOH ratio was maintained at 2 and CFA/Alkali Activator (AA) was maintained at 2.5.

For the development/synthesis of lightweight geopolymeric materials, the second raw material which was waste paper was added in order to achieve the aim of this study. Therefore, the optimisation of the amount of waste paper was carried out as shown below.

3.5.1.2. Waste-Cardboard/Pulp content

The alkali liquid consisted of sodium hydroxide and sodium silicate solution. The optimisation of the additional quantities of waste cardboard paper/pulp (0.30, 0.60, 0.90 and 1.20 kg of waste cardboard paper) added to the geopolymerisation process is described as follows.

In this section, the explanation is given for the experiments carried out using 12 M of NaOH only because the same procedure was followed for the concentration of 10 and 14 M of NaOH.

Note: In the present study, all formulations and conditions for the synthesis of geopolymer materials were performed in triplicate.

0.30 kg of waste cardboard paper content (10%)

In the first trial, 2.7 kg of CFA was mixed with 0.36 kg (12 M) of NaOH and 0.72 kg of Na_2SiO_3 was mixed for 20 minutes. 0.30 kg (10 %) of cardboard waste paper was added to the mixture and mixed for 25 minutes. The paste was cast into hydraulic press moulds (100mm x 100mm x

130mm). The desired hydraulic pressure was selected and the geopolymer paste was compressed into a 100mm³ cube. The geopolymer block was demoulded and covered in plastic in order to keep the moisture in the samples and the moulds were put in an oven at 60°C. After drying and hardening of the geopolymer, the samples were stored in plastic at room temperature and tested at the age of 7 days for compressive strength, water absorption, shrinkage, and tensile strength. The specimen was named B1SH12WH0.35 in Table 3.5-2.

In the second trial, the experiment was carried out as in the first trial, except that the moulds were put in an oven at 80°C. The specimen was named B2SH12WH0.35 in Table 3.5-2.

0.60 kg of waste cardboard paper content (20%)

In the first trial, 2.4 kg of CFA was mixed with 0.32 kg (12 M) NaOH of 12 M and 0.64 kg of Na₂SiO₃ was mixed for 20 minutes. 0.60 kg (20%) of cardboard waste paper was added to the mixture and mixed for 25 minutes. The paste was cast into hydraulic press moulds (100mm x 100mm x 130mm). The desired hydraulic pressure was selected and the geopolymer paste was compressed into a 100mm³ cube. The block was demoulded and covered in plastic in order to keep the moisture in the samples and the moulds were put in an oven at 60 C. After the geopolymer was dried and hardened, samples were stored in plastic at room temperature and tested at the age of 7 days for tests such as compressive strength, water absorption, shrinkage, and tensile strength. The specimen was named C1SH12WH0.35 in Table 3.5-3.

In the second trial, the same experimental procedure was followed as in the first trial and then the geopolymer paste was cured in an oven at 60°C. However, in this trial, the geopolymer paste was cured in an oven at 80°C and tested at the age of 7 days for compressive strength. These samples were symbolised by C2SH12WH0.35 in Table 3.5-3.

0.90 kg of waste cardboard paper content (30%)

In the first trial, 2.10 kg of CFA was mixed with 0.28 kg (12 M) of NaOH 12 M and 0.56 kg of Na₂SiO₃ for 20 minutes. 0.90 kg (30 per cent) of cardboard waste paper was added to the mixture and mixed for 25 minutes. The paste was cast into hydraulic press moulds (100mm x 100mm x 130mm). The desired hydraulic pressure was selected and the geopolymer paste was compressed into a 100mm³ cube. The block was demoulded and covered in plastic to keep the moisture in the samples and the moulds were put in an oven at 60°C. After the geopolymer is dry and hardened, samples were stored in plastic at room temperature and tested at the age

of 7 days for tests such as compressive strength, water absorption, shrinkage, and tensile strength. The specimen was named D1SH12WH0.35 in Table 3.5-4.

In the second trial, the same experimental procedure was followed as in the first trial when the geopolymer paste was cured in an oven at 60°C. However, in this trial, the geopolymer paste was cured in an oven at 80°C and tested at the age of 7 days for compressive strength. These samples were symbolised by D2SH12WH0.35 in Table 3.5-4.

1.20 kg of waste cardboard paper content (40%)

In the first trial, 1.80 kg of CFA was mixed with 0.24 kg of NaOH of 12 M and 0.48 kg of Na₂SiO₃ for 20 minutes. 1.20 kg (40%) of cardboard waste paper was added to the mixture and mixed for 25 minutes. The paste was cast into hydraulic press moulds (100mm x 100mm x 130mm). The desired hydraulic pressure was selected and the geopolymer paste was compressed into a 100mm³ cube. The block was demoulded and covered in plastic to keep the moisture in the samples and the moulds were put in an oven at 60°C. After the geopolymer was dry and hardened, samples were stored in plastic at room temperature and tested at the age of 7 days for tests such as compressive strength, water absorption, shrinkage, and tensile strength. The specimen was named E1SH12WH0.35 in Table 3.5-5.

In the second trial, the same experimental procedure was followed as in the first trial when the geopolymer paste was cured in an oven at 60°C. However, in this trial, the geopolymer paste was cured in an oven at 80°C and tested at the age of 7 days for compressive strength. These samples were symbolised by E2SH12WH0.35 in Table 3.5-5.

3.5.1.3. Water Content

The alkali liquid consisted of sodium hydroxide and sodium silicate solution. The optimisation of the additional quantities of waste-cardboard paper and CFA to water was investigated and varied as follows: 0.90 kg, 1.80 kg, 2.70 kg and 3.60 kg water were added to the geopolymerisation process or the ratio of dry solids to water was 0.3, 0.6, 0.9, 1.2. The process was described as follows.

The pulp was received from the supplier with 73 to 75% water. All waste paper content percentages contained the same amount of water. However, as waste paper content increased, so did water content. As noted in Chapter 2, water does have an effect on geopolymer

compressive strength, therefore it was important to monitor the amount added to the system. To keep the water added to the system constant, two measures were taken: either the water in pulp was extracted or added to the system, depending on the amount of water being investigated.

Using 0.90 kg of water

In this section, the explanation is given for the experiments carried out using 10 M of NaOH only and 30% waste paper content which is 0.90 kg because the same procedure was followed for the concentration of 12 and 14 M of NaOH and 10%, 20% and 40% waste cardboard paper.

In the case of 10% waste paper content, the pulp contained 0.9 kg of water based on the ratio of water to waste paper, meaning when investigating water at 0.9 kg, no water was added or extracted.

In the case of 20% waste paper content, the pulp contained 1.80 kg of water which means that half of the water had to be extracted in order to maintain water content of 0.90 kg.

In the case of 30% waste paper content, the pulp contained 2.70 kg of water and this means more than half of the water had to be extracted in order to maintain the 0.90 kg water content. Waste paper was weighed and the water was drained until the water content of the pulp was 0.90 kg.

The experiment started by taking 0.28 kg NaOH of 12M and 0.56kg Na_2SiO_3 and 2.10 kg CFA. The paste was mixed for 20 minutes altogether before 0.9 kg (30 wt%) of waste paper containing 0.9 kg of water was added and mixed for 25 minutes to ensure homogeneity of the paste. The paste was cast into the compression moulds (100mm x 100 mm x 150 mm). The geopolymer paste was compressed at a specific pressure to a 100 mm³ geopolymer block. The block was covered with a plastic sheet to keep the moisture in the samples and the moulds were put in an oven at 80°C for 60 hours. Then, the hardened geopolymer samples were stored in plastic at room temperature and tested at the age of 7 days for compressive strength. These samples were symbolised by WP30SH10T80 in Table 3.6-1. In the second trial, the same experimental procedure was followed as in the first trial, except that in this trial the temperature was 60°C and it took 96 hours for the geopolymer to harden in the oven hardened geopolymer. These samples were symbolised by WP30SH10T60 in Table 3.6-1.

Using 1.80 kg of water

In this section, the explanation is given for the experiments carried out using 10 M of NaOH only and 30% waste paper content which is 0.90 kg because the same procedure was followed for the concentration of 10 and 14 M of NaOH and 10%, 20% and 40% waste cardboard.

In the case of 10% waste paper content, the pulp contained 0.9 kg of water based on the ratio of water to waste paper, meaning to maintain 1.80 kg, 0.90 kg of water had to be added to the system.

In the case of 20% waste paper content, the pulp contained 1.80 kg of water which means no water had to be added to the system.

In the case of 30% and 40 % waste paper content, the pulp contained 2.70 kg and 3.60 kg of water respectively, which means that about 0.90 kg and 1.80 kg of water had to be extracted respectively in order to maintain the 1.80 kg water content. Waste paper was weighed and dried at 60°C until the water content of the pulp was 1.80 kg.

The experiment started by taking 0.28 kg NaOH of 10M and 0.56 kg Na_2SiO_3 , 0.90 kg waste-cardboard paper and 2.10 kg CFA. The paste was mixed for 20 minutes altogether before 0.9 kg (30 wt%) of waste paper containing 1.80 kg of water was added and mixed for 25 minutes to ensure homogeneity of the paste. The paste was cast into the compression moulds (100 mm x 100 mm x 150 mm). The geopolymer paste was compressed at a specific pressure into a 100 mm³ geopolymer block. The block was covered with a plastic sheet to keep the moisture in the samples and the moulds were put in an oven at 80°C for 60 hours. Then the hardened geopolymer samples were stored in plastic at room temperature and tested at the age of 7 days for compressive strength. These samples were symbolised by WP30SH10T80 in Table 3.6-1. In the second trial, the same experimental procedure was followed as in the first trial, except that in this trial the temperature was 60°C and it took 96 hours for the geopolymer to harden in the oven. These samples were symbolised by WP30SH10T60 in Table 3.6-1.

Using 2.70 kg of water

In this section, the explanation is given for the experiments carried out using 10 M of NaOH only and 30% waste paper content which is 1.35 kg because the same procedure was followed for the concentration of 10 and 14 M of NaOH and 10%, 20% and 40% waste cardboard.

In the case of 10% waste paper content, the pulp contained 0.90 kg of water based on the ratio of water to waste paper, meaning that to maintain 2.70 kg, 1.80 kg of water had to be added to the system.

In the case of 20% waste paper content, the pulp contained 1.80 kg of water, meaning that to maintain 2.70 kg, 0.90 kg of water had to be added to the system

In the case of 30%, waste paper pulp contained 2.70 kg of water already, meaning that there was no need to add water to the system.

In the case of 40 % waste paper content, the pulp contained 3.60 kg of water which means that about 0.90 kg of water had to be extracted in order to maintain 2.70 kg water content. Waste paper was weighed and dried at 60 degrees Celsius until the water content was 2.70 kg in the pulp.

The experiment started by taking 0.28 kg NaOH of 12M and 0.56 kg Na_2SiO_3 , 0.90 kg waste-cardboard and 2.10 kg CFA. The paste was mixed for 20 minutes altogether before 0.9 kg (30 wt%) of waste paper containing 2.70 kg of water was added and mixed for 25 minutes to ensure homogeneity of the paste. The paste was cast into the compression moulds (100mm x 100 mm x 150 mm). The geopolymer paste was compressed at a specific pressure to a 100 mm³ geopolymer block. The block was covered with a plastic sheet to keep the moisture in the samples and the moulds were put in an oven at 80 °C for 60 hours. Then the hardened geopolymer samples were stored in plastic at room temperature and tested at the age of 7 days for compressive strength. These samples were symbolised by WP30SH10T80 in Table 3.3.

In the second trial, the same experimental procedure was followed as in the first trial, except that the temperature was 60 °C and it took 96 hours for the geopolymer to harden in the oven. These samples were symbolised by WP30SH10T60 in Table 3.5-1.

Using 3.60 kg of water

In this section, the explanation is given for the experiments carried out using 10 M of NaOH only and 30% waste paper content which is 0.90 kg because the same procedure was followed for the concentration of 10 and 14 M of NaOH and 10%, 20% and 40% waste cardboard paper.

In the case of 10% waste paper content, the pulp contained 0.90 kg of water based on the ratio of water to waste paper, meaning that to maintain 3.60 kg, 2.70 kg of water had to be added to the system.

In the case of 20% waste paper content, the pulp contained 1.80 kg, meaning to maintain 3.60 kg, 1.80 kg of water had to be added to the system

In the case of 30%, waste paper, the pulp contained 2.70 kg of water already meaning to maintain 3.60 kg, 0.90 kg of water had to be added to the system

In the case of 40 % waste paper content, the pulp contained 3.60 kg of water, therefore no water had to be extracted or added to the system.

The experiment started by taking 0.42kg NaOH of 12M and 0.28 kg Na_2SiO_3 , 0.56 kg waste-cardboard paper and 2.10 kg CFA. The paste was mixed for 20 minutes altogether before 0.9 kg (30 wt.%) of waste paper containing 3.60 kg of water was added and mixed for 25 minutes to ensure homogeneity of the paste. The paste was cast into the compression moulds (100mm x 100 mm x 150 mm). The geopolymer paste is compressed at a specific pressure to a 100 mm³ geopolymer block. The block was covered with a plastic sheet to keep the moisture in the samples and the moulds were put in an oven at 80°C for 60 hours. Then the hardened geopolymer samples were stored in plastic at room temperature and tested at the age of 7 days for compressive strength. These samples were symbolised by WP30SH10T80 in Table 3.6-1.

In the second trial, the same experimental procedure was followed as in the first trial, except that in this trial the temperature was 60°C and it took 96 hours for the geopolymer to harden in the oven. These samples were symbolised by WP30SH10T60 in Table 3.6-1.

3.5.1.4. Curing Temperature

The formulations used resulted in the generation of geopolymer pastes which were cured in an oven at two temperatures as described below:

60°C

All the geopolymer pastes produced using the formulations developed in this study were cured in an oven at 60°C. Curing time was different for geopolymers with 0 % waste cardboard which cured for 24 hours whilst 10 % to 40 % waste cardboard content cured for 80 hours.

80 C

All the geopolymer pastes produced using the formulations developed in this study were cured in an oven at 80°C. Curing time was different for geopolymers with 0 % waste cardboard which cured for 24 hours whilst 10 % to 40 % waste cardboard content cured for 60 hours. After curing the geopolymer paste for all the formulations developed in this study in the oven for 24 hours at either 60 or 80°C the samples/specimens were aged for 7 days before characterisation.

Development and study of fly ash-based geopolymer has been done extensively at Environmental Nano-Science. The method of synthesising geopolymer was established and several publications on the development of fly ash-based geopolymer were produced. The reinforcement of other materials as described in Chapter 2 has enhanced the properties of geopolymer for specific purposes. However, despite the extensive study on geopolymer manufacturing, reinforcing waste paper to fly ash-based geopolymer has been done in different ways by a few authors. Most researchers have used waste paper after incineration. Aryomi (2016) did not make waste paper ash but dried it into fine aggregated form in order to minimise the water intake during manufacturing. Aryomi (2016) used newspaper as waste paper and pulverised the it before drying it for four days. Based on the aforementioned studies, the current study did not incinerate the paper as this required more energy.

Tables 3.5-1 to 3.5-5 represents different formulations, and the best formulation and conditions were determined by material characterisation.

A refers to 0% waste paper content; B refers to 10% waste paper content; C refers to 20% waste paper content, D refers to 30% waste paper content and E refers to 40% waste paper content.

1 and 2 stand for 60°C and 80°C curing temperature respectively.

SH refers to sodium hydroxide concentration.

W refers to solid to liquid ratio (CFA & WP/H₂O)

Table 3.5-1: The mixture proportions and conditions used in the synthesis of lightweight geopolymeric material with 0 % waste paper content

Specimen ID	S/L	WP Content %	Curing Temperature	Aging Days	CFA/AA	SS/SH	Constant Parameters
A1SH10W3.33	3.33	0	60	7	2.5	2	Sodium hydroxide concentration is 10 M. Mixing time is 45 minutes. Curing time is 24 hours
A1SH10W1.67	1.67						
A1SH10W1.11	1.11						
A1SH10W0.83	0.83						
A2SH10W3.33	3.33	0	80	7	2.5	2	
A2SH10W1.67	1.67						
A2SH10W1.11	1.11						
A2SH10W0.83	0.83						
A1SH12W3.33	3.33	0	60	7	2.5	2	Sodium hydroxide concentration is 12 M. Mixing time is 45 minutes. Curing time is 24 hours
A1SH12W1.67	1.67						
A1SH12W1.11	1.11						
A1SH12W0.83	0.83						
A2SH12W3.33	3.33	0	80	7	2.5	2	
A2SH12W1.67	1.67						
A2SH12W1.11	1.11						
A2SH12W0.83	0.83						
A1SH14W3.33	3.33	0	60	7	2.5	2	Sodium hydroxide concentration is 14 M. Mixing time is 45 minutes. Curing time is 24 hours
A1SH14W1.67	1.67						
A1SH14W1.11	1.11						
A1SH14W0.83	0.83						
A2SH14W3.33	3.33	0	80	7	2.5	2	
A2SH14W1.67	1.67						
A2SH14W1.11	1.11						
A2SH14W0.83	0.83						

The specimen ID

A represents waste paper content 0 %; 1 and 2 represent the temperature 60°C and 80°C respectively; SH is the sodium hydroxide concentration; W represents the solid to water ratio.

Table 3.5-2: The mixture proportions and conditions used in the synthesis of lightweight geopolymeric material with 10 % waste paper content

Specimen ID	S/L	WP Content %	Curing Temperature	Aging Days	CFA/AA	SS/SH	Constant Parameters	
B1SH10W3.33	3.33	10	60	7	2.5	2	Sodium hydroxide concentration is 10 M. Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C	
B1SH10W1.67	1.67							
B1SH10W1.11	1.11							
B1SH10W0.83	0.83							
B2SH10W3.33	3.33	10	80	7	2.5	2		
B2SH10W1.67	1.67							
B2SH10W1.11	1.11							
B2SH10W0.83	0.83							
B1SH12W3.33	3.33	10	60	7	2.5	2		Sodium hydroxide concentration is 12 M. Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
B1SH12W1.67	1.67							
B1SH12W1.11	1.11							
B1SH12W0.83	0.83							
B2SH12W3.33	3.33	10	80	7	2.5	2		
B2SH12W1.67	1.67							
B2SH12W1.11	1.11							
B2SH12W0.83	0.83							
B1SH14W3.33	3.33	10	60	7	2.5	2	Sodium hydroxide concentration is 14 M. Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C	
B1SH14W1.67	1.67							
B1SH14W1.11	1.11							
B1SH14W0.83	0.83							
B2SH14W3.33	3.33	10	80	7	2.5	2		
B2SH14W1.67	1.67							
B2SH14W1.11	1.11							
B2SH14W0.83	0.83							

The specimen ID

B represents waste paper content 10%; 1 and 2 represent the temperature 60°C and 80°C respectively; SH is the sodium hydroxide concentration; WH represents the waste paper to water ratio.

Table 3.5-3: shows the mixture proportions and conditions used in the synthesis of lightweight geopolymeric material with 20 % waste paper content

Specimen ID	WP/H ₂ O	WP Content %	Curing Temperature	Aging Days	CFA/AA	SS/SH	Constant Parameters
C1SH10W3.33	3.33	20	60	7	2.5	2	Mixing time is 45 minutes. curing time is 80 hours for 60°C and 60 hours for 80°C
C1SH10W1.67	1.67						
C1SH10W1.11	1.11						
C1SH10W0.83	0.83						
C2SH10W3.33	3.33	20	80	7	2.5	2	
C2SH10W1.67	1.67						
C2SH10W1.11	1.11						
C2SH10W0.83	0.83						
C1SH12W3.33	3.33	20	60	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
C1SH12W1.67	1.67						
C1SH12W1.11	1.11						
C1SH12W0.83	0.83						
C2SH12W3.33	3.33	20	80	7	2.5	2	
C2SH12W1.67	1.67						
C2SH12W1.11	1.11						
C2SH12W0.83	0.83						
C1SH14W3.33	3.33	20	60	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
C1SH14W1.67	1.67						
C1SH14W1.11	1.11						
C1SH14W0.83	0.83						
C2SH14W3.33	3.33	20	80	7	2.5	2	
C2SH14W1.67	1.67						
C2SH14W1.11	1.11						
C2SH14W0.83	0.83						

The specimen ID

C represents waste paper content 20 %; 1 and 2 represent the temperature 60°C and 80°C respectively; SH is the sodium hydroxide concentration; WH represents the waste paper to water ratio.

Table 3.5-4: shows the mixture proportions and conditions used in the synthesis of lightweight geopolymeric material with 30 % waste paper content.

Specimen ID	WP/H ₂ O	WP Content %	Curing Temperature	Aging Days	CFA/AA	SS/SH	Constant Parameters
D1SH10W3.33	3.33	30	60	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
D1SH10W1.67	1.67						
D1SH10W1.11	1.11						
D1SH10W0.83	0.83						
D2SH10W3.33	3.33	30	80	7	2.5	2	
D2SH10W1.67	1.67						
D2SH10W1.11	1.11						
D2SH10W0.83	0.83						
D1SH12W3.33	3.33	30	60	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60 C and 60 hours for 80°C
D1SH12W1.67	1.67						
D1SH12W1.11	1.11						
D1SH12W0.83	0.83						
D2SH12W3.33	3.33	30	80	7	2.5	2	
D2SH12W1.67	1.67						
D2SH12W1.11	1.11						
D2SH12W0.83	0.83						
D1SH14W3.33	3.33	30	60	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
D1SH14W1.67	1.67						
D1SH14W1.11	1.11						
D1SH14W0.83	0.83						
D2SH14W3.33	3.33	30	80	7	2.5	2	
D2SH14W1.67	1.67						
D2SH14W1.11	1.11						
D2SH14W0.83	0.83						

The specimen ID

D represents waste paper content 30 %; 1 and 2 represent the temperature 60°C and 80°C respectively; SH is the sodium hydroxide concentration; WH represents the waste paper to water ratio.

Table 3.5-5: shows the mixture proportions and conditions used in the synthesis of lightweight geopolymeric material with 40 % waste paper content

Specimen ID	WP/H ₂ O	WP Content %	Curing Temperature	Aging Days	CFA/AA	SS/SH	Constant Parameters
E1SH10W3.33	3.33	40	80	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
E1SH10W1.67	1.67						
E1SH10W1.11	1.11						
E1SH10W0.83	0.83						
E2SH10W3.33	3.33	40	80	7	2.5	2	
E2SH10W1.67	1.67						
E2SH10W1.11	1.11						
E2SH10W0.83	0.83						
E1SH12W3.33	3.33	40	60	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
E1SH12W1.67	1.67						
E1SH12W1.11	1.11						
E1SH12W0.83	0.83						
E2SH12W3.33	3.33	40	80	7	2.5	2	
E2SH12W1.67	1.67						
E2SH12W1.11	1.11						
E2SH12W0.83	0.83						
E1SH14W3.33	3.33	40	60	7	2.5	2	Mixing time is 45 minutes. Curing time is 80 hours for 60°C and 60 hours for 80°C
E1SH14W1.67	1.67						
E1SH14W1.11	1.11						
E1SH14W0.83	0.83						
E2SH14W3.33	3.33	40	80	7	2.5	2	
E2SH14W1.67	1.67						
E2SH14W1.11	1.11						
E2SH14W0.83	0.83						

The specimen ID

E represents waste paper content 40%; 1 and 2 represent the temperature 60°C and 80°C respectively; SH is the sodium hydroxide concentration; WH represents the waste paper to water ratio.

3.6. Procedure for lightweight geopolymeric material manufacturing

Manufacturing lightweight geopolymeric materials requires three easy steps, which will be described in detail in the following sections.

Preparation of Liquids

Sodium hydroxide (NaOH) flakes were used in this experiment. NaOH flakes were dissolved in water to make an alkali solution. In order to reduce the use of chemicals in manufacturing, only three concentrations (10 molar (M), 12 M and 14 M) were used. For example, NaOH solution with a concentration of 10 M consisted of $10 \times 40 = 400$ grams of NaOH solids (in flake form) per litre of solution, where 40 is the molecular weight of NaOH. The mass of the NaOH solids was only a fraction of the mass of the NaOH solution, and water was the main component. The NaOH solution was prepared at least 24 hours before the experiment, the NaOH reaction and the liquid are exothermic, therefore the 24 hour cycle allows it to cool down before being used for the test.

The sodium silicate solution was blended together with the NaOH solution according to the proportions laid down in tables 3.5-1 to 3.5-5 for the preparation of the alkaline water. On the day the specimens were cast, the alkaline liquid was mixed together with the extra water (if any) to prepare the liquid component of the mixture.

Pulp Preparation

Water has a significant impact on geopolymer mechanical properties therefore, it had to be minimised when manufacturing geopolymer. In order to know the amount of water in the pulp, three pulp samples with the weight of 5 g were weighed before and dried till the mass of the pulp was constant and the average water content was calculated. The percentage of water in pulp was approximately 75%. The pulp was collected from a recycling company as mentioned above, therefore some of the granules were lumpy. Prior to preparing the paste, the pulp was weighed based on the ratio required for this experiment; the ratio of waste paper to CFA was from 0.1 to 0.4, and 75% of the water in pulp was considered. The pulp was weighed and sorted before being mixed with the geopolymer paste.

Manufacture of Fresh Concrete and Casting

The procedure of making geopolymer paste was simple. The CFA was mixed with an alkaline solution in an Imer laboratory mixer for about 20 minutes. Pulp was added to the mixture for 25 minutes until the paste was well blended. The fresh paste was then cast into the moulds. The compaction method used for casting was a hydraulic press, used to avoid shrinkage and implosion of geopolymers whilst curing in the oven.

Curing of Test Specimens

After casting, the test specimens were covered with plastic film to minimize water evaporation during curing at an elevated temperature. In the current study, one type of curing was used which was dry curing. The specimens without waste paper content were heat-cured at 60°C and 80 °C for 24 hours (Hardjito et. al., 2004). The specimens with waste paper were subjected to a different procedure.

The specimens with waste paper content were cured for 24 hours at 80°C and 48 hours at 60°C to allow the paste to set. Thereafter specimens were demoulded and weighed then returned to the oven at 60°C and 80°C covered with a plastic film. The plastic film was used in order to minimise fast evaporation in order to allow the geopolymerisation reaction to occur. After every 24 hours, the specimens were weighed until 60 hours for 80°C and 92 for 60°C where the weight of the specimen did not show significant change. Upon demoulding, the samples were left to air dry at room temperature in the laboratory until the day of the experiment.

The formulations of each sample coded from Table 3.5-1 to Table 3.5-5 were prepared by mixing CFA and waste cardboard paper pulp with a solution of alkali liquids to produce lightweight geopolymeric material. The alkaline liquid/alkaline actuator consisted of sodium hydroxide and sodium silicate. CFA was mixed in the mixing unit made of a plastic tank and a mechanical stirrer rotating at a speed of 55 rpm for 20 minutes in order to form a homogeneous paste; waste cardboard pulp was added and mixed for an extra 25 minutes, resulting in a total of 45 minutes mixing time. Figure 3.6-1 shows the schematic block flow diagram (BFD) route followed during the synthesis of lightweight geopolymeric material and characterised using the analytical techniques, durability and mechanical properties described in section 3.7.

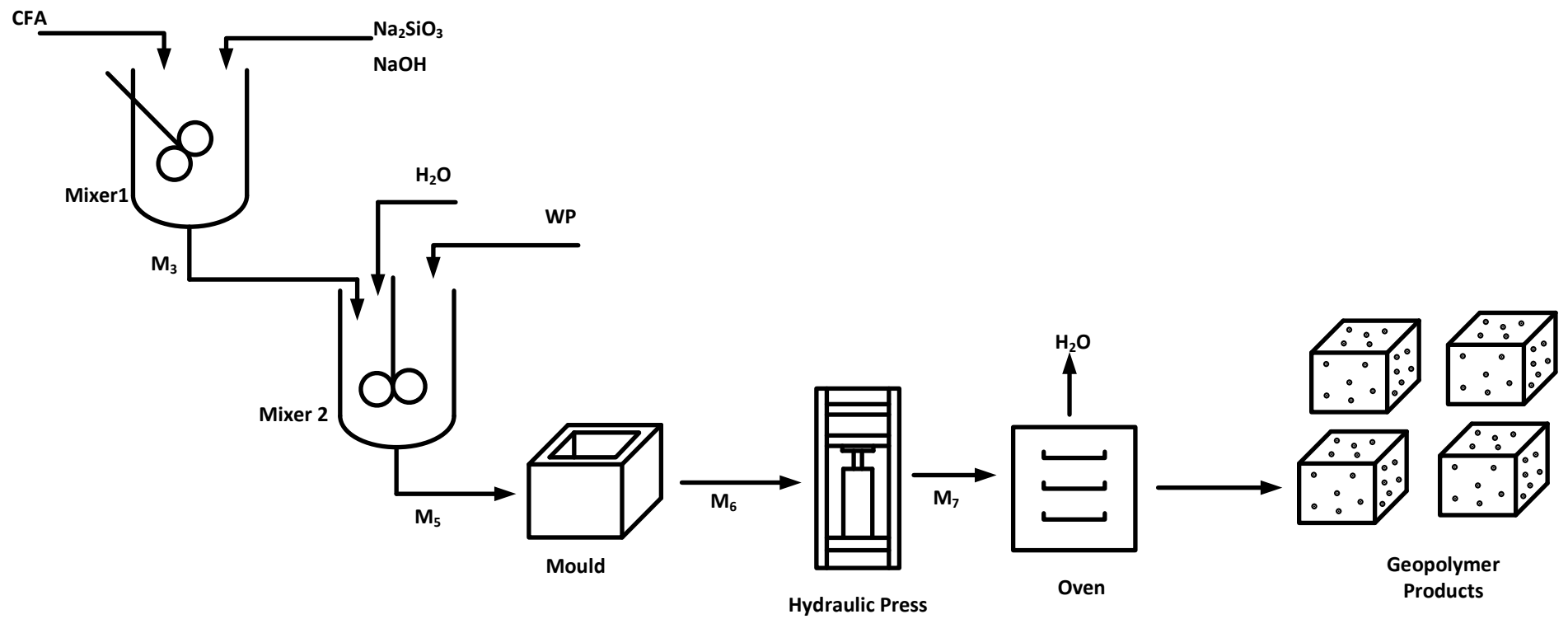


Figure 3.6-1: PFD of the lightweight geopolymeric material synthesis

Figure 3.6-1 shows the experimental setup used in the geopolymerisation process. How the geopolymer paste formed as described in section 3.3.1.

3.7. Analytical Techniques

The different analytical techniques used to analyse raw materials (solid samples) in this study are presented in the following sub-sections.

3.7.1. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is an analytical method used to determine the morphology and crystalline structure of the sample. The electrons communicate with the atoms in the sample, producing different signals that contain information on the surface and composition of the sample. The electron beam is scanned in a raster scan pattern and the position of the beam is combined with the detected signal to create the image. The most popular SEM mode is the detection of secondary electrons released by electron beam-excited atoms (Stokes, 2008).

The specimens were analysed using a Hitachi X-650 Scanning Electron Micro-analyzer fitted with a 25 kV CDU-lead detector and a tungsten filament. The specimens were prepared by sticking a carbon adhesive tape to the aluminium stub. A small amount of each sample was added to the carbon adhesive tape, which was coated with carbon in a K950X carbon evaporator for 6 seconds to make it conductive. Samples were placed in the column of the SEM and the samples were examined under various magnifications. Micrographs of each sample were captured and displayed on an LCD computer.

3.7.2. X-ray Diffraction (XRD)

Qualitative X-ray diffraction (XRD) was used to identify and quantify the mineral phases existing in the waste cardboard pulp and CFA and different waste cardboard paper content geopolymer materials produced, in the ground form. XRD research was conducted on solid samples such as coal fly ash and lightweight geopolymers for all paper waste (0 per cent, 10 per cent, 20 per cent, 30 per cent and 40 per cent). A Philips PANalytical instrument with a pw3830 X-ray generator operated at 40 kV and 25 mA was used for XRD research. In order to carry out XRD analysis, approximately 0.5 g of the sample was prepared and placed onto a glass substrate inside a hollow sample holder. The sample height was levelled with respect to the edge of the sample holder and inserted into the XRD instrument. The analysis was carried out between 0°

and 90° and the obtained mineral phases were identified using HighScore Xpert software. The spectra obtained were compared with the standard diffraction powder database patterns provided by the International Center for Diffraction Data (ICDD).

Every single sample was oven-dried at 105° C for 12 hours to remove any adsorbed moisture and ground from the powder prior to analysis. X-ray diffraction is based on the constructive interaction of the monochromatic X-ray and the crystalline specimen. The interaction of the incident rays with the sample creates positive interference (and diffraction) when conditions comply with Bragg's Law;

$$n\lambda=2d\sin\theta$$

Where: n is a positive integer; λ is the wavelength of the incident; θ is the scattering angle and d is the interplanar distance.

This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing of a crystalline specimen. Such diffraction X-rays are then detected, analyzed and counted. Through scanning the specimen through a spectrum of 180 degree angles, due to the random orientation of the powdered material, all possible diffraction directions of the lattice should be obtained. The conversion of the diffraction peaks to the d-spaces allows the mineral to be identified because each mineral has a set of unique d-spaces. This is usually achieved by comparing the d-spaces with the standard reference patterns.

3.7.3. X-ray Fluorescence

The X-Ray Fluorescence (XRF) spectrometer is an x-ray instrument widely used to identify chemical elements in the liquid phase, solid phase or powder phase (Hamood, 2013). The complicated operating principle of the XRF spectrometer requires the release of some tightly held electrons by energy absorption, which leads to the fragmentation of the atoms and the eventual substitution of some missing inner electrons with outer electrons. Following a couple of other processes that take place within the device, the elements found in the samples tested are detected by fluorescent X-rays. The bulk chemical composition of the dry components of the CFA specimen were analysed using the X-Ray fluorescence (XRF) spectrometer.

Every specimen was oven-dried for 12 hours at 105° C to extract adsorbed water prior to analysis; the samples were crushed to a particle size of fewer than 100 μm with a jaw crusher

and then milled in a tungsten zib mill to avoid trace and REE contamination prior to preparation of a fused disk for major and trace element analysis. Glass disks were prepared for XRF analysis using 10 g of high purity trace element and REE free flux element ($\text{LiBO}_2 = 80$ per cent, $\text{Li}_2\text{B}_4\text{O}_7 = 20$ per cent) combined with 1 g of the sample. The composition of the main elements were calculated by the Philips 1404 Wavelength Dispersive Spectrometer.

The setup was as follows: the instrument was fitted with a gas-flow proportional counter (a gas mixture of 90% Ar and 10% CH_4) and a scintillation detector. The study of the main elements was carried out on a fused glass disk at 50 mA and 50 kV tube operating conditions. The matrix effects in the samples were corrected by adding theoretical alpha factors and calculated line overlap factors to the raw intensities measured with the SuperQ Philips programme.

3.8. Engineering Properties

Construction materials are tested for properties that affect/influence their performance when applying the material in buildings. Based on literature and recommendations from the South African National Standards and the American Society for Testing and Materials (ASTM), properties of lightweight geopolymeric bricks to be applied in non-load bearing applications were tested for the following tests: compressive strength, water absorption, shrinkage, and flammability. Detailed results for each of the properties tested are presented and discussed in Chapter 4 of this dissertation. In addition, more additional experiments were conducted to examine the effects of hydraulic pressure on the shrinkage of the geopolymeric product. Having obtained an optimum mixture composition for lightweight geopolymeric material, test samples in the form of 100 mm x 100 mm x 100 mm cubes were developed to determine its various engineering properties.

3.8.1. Compressive Strength Test

For each series of tests, a set of standard size cubes were made. The size of the cubes was 100 mm length by 100 mm width or 100 mm height. The cubes were tested for compression strength based on South African National Standards SANS 1012.91999. Compressive strength is characterized as the ability of the material, in this case, lightweight geopolymeric material, to resist breakage when exposed to the axially applied load, whether on the edge or on the side of the block. Mathematically, compressive strength is expressed as the ratio of crushing load (N) to the surface area (mm^2) where the load has been applied. The compressive strength

test was applied on all specimens at 7 days of curing. Compressive strength tests were performed using the SANS method 5863:2006 standard procedure. The equipment used was a PTL-10 model MTS Criterion™ Series 60 (60 ton) Standard: Modified ASTM 370 Rate: 10 mm/min cubic samples. The compressive strength machine used in this study is depicted in Figure 3.8-1.

The geometry of the sample was determined using a vernier calliper, and a laboratory balance was used to weigh the mass of the block. The volume of the geopolymers block produced was $100 \times 100 \times 100 \text{ mm}^3$ and the weight varied based on the geopolymers waste cardboard content. The specimens were placed between two compression plates (one plate at the bottom and the other on top). The specification for the compressive strength of non-load bearing blocks differs for different codes, countries and for different applications. BS 771-4:2011, which is one of the EN771 series of standards for masonry blocks, stipulates a minimum strength of 1.5 MPa for lightweight non-load bearing blocks to be used for partitioning /non-structural applications. The Ghana Building Code (National Committee 1989) specifies a minimum of 1.4 N/mm² (max) for non-loading walls.

The compressive force was calculated by dividing the crushing load by the loaded area A (mm²). The average of the 3 specimen samples tested for each mixture was 0.01 N/mm². Three samples were prepared for 7 days and tested for compressive strength. The compressive strength was calculated from the applied load at the cube failure point. The average of the three cube tests was reported. The calculation sample is reported in the Appendix A to this study.



Figure 3.8-1: Compressive strength test machine.

3.8.2. Water Absorption

Water absorption can be characterized as a phenomenon in which the material, due to the intermolecular attraction within the liquid and the solid, absorbs liquid into the small opening within its microstructure. Organic materials, including bricks, blocks, tiles, mortar, and concrete, are prone to water absorption and to capillary action due to their porosity and permeability (Hall & Hoff, 2011; Karagiannis et al., 2016). It is recommended to determine the water absorption test on lightweight geopolymeric material as it is manufactured with waste paper which is known as a very high water absorber. This analysis assisted with determining whether this lightweight geopolymeric material can be applied in the construction industry as it is already unfit for weather exposure.

The size of the specimen used was 100mm x 100mm x 100mm, dried to a constant mass at 80 degrees Celsius and the specimen was cooled at ambient temperature for 24 hours. The specimen dimensions were measured. A container was filled with room temperature tap water.

The specimen was weighed before immersion in water. In literature the specimen is usually immersed in water for 24 hours; however, for this research, the samples were immersed in water for a maximum time of 30 minutes only. After the immersion, the specimen was wiped to remove surface water and weighed to determine the water that has been absorbed over the specific period. The equation below was used to determine the percentage of water absorption. Six blocks were used for each waste paper content. The geopolymer blocks were immersed in water for 5 minutes, 10 minutes, 15 minutes, 20 minutes, 25 minutes and 30 minutes.

To calculate the amount of water absorbed by the specimen, the following formula was used,

$$W_{ab} = M_f - M_i$$

The formula used to determine the water absorption rate follows:

$$W_{abrate} = \frac{M_f - M_i}{t}$$

Where:

M_f is final mass recorded after immersion in kg

M_i is initial mass before immersion in kg

t is the time of immersion in minutes

W_{ab} is the total amount of water absorbed in kg

W_{abrate} is the water absorption rate

3.8.3. Drying Shrinkage Test

Drying shrinkage is one of the important engineering properties to be considered for the production of lightweight geopolymeric materials. Shrinkage is a drop in the amount of concrete with a time reduction that is independent of external behavior to the concrete. According to the literature, the raw materials used in manufacturing geopolymeric materials have a direct impact on drying shrinkage as these materials will determine the amount of water

used whilst mixing the paste. The amount of water used/added to the process was evaporated during curing, and loss of water would result in shrinkage of the specimen.

Test specimens for drying shrinkage tests were 100 mm x 100 mm x 100 mm cubes. The cubes were measured by a calliper as the shrinkage was macro rather than at the micro level, which would require the use of studs. The shrinkage measurements started on the second day after the concrete was cast. On the first day after casting, the specimens were removed from moulds and the first measurement was taken. The next measurement was on the third day of the casting, which was considered Day 1 for the measurement of the drying shrinkage. The measurements continued every day during the first week of treatment, once a week until the seventh day when the specimen was ready for the compressive strength test.

The shrinkage percentage was calculated based on the equation below:

$$\text{Drying Shrinkage \%} = \frac{V_1 - V_2}{V_1} \times 100$$

Where: V_1 is the initial volume of the geopolymer

V_2 is the final volume of the geopolymer

3.8.4. Density Test

The calculation of how many particles of an element are pushed into a given space is called density. For this study, density was of interest as the shrinkage test was done (Averill and Elderedge., 2007). The density test was analysed to determine the relationship between waste paper and density. Density is mathematically calculated by dividing the mass (kg) of the geopolymeric block by the volume (m^3) of the block. The more closely packed particles in a given space, the higher the density. BS EN 771-4:2011 states that the net density of lightweight non-load bearing blocks is typically between 300 kg/m^3 and 1000 kg/m^3 . BS EN 771-4:2011 recommends a range of 300 kg/m^3 to 1000 kg/m^3 for lightweight non-load bearings and a minimum of 625 kg/m^3 for lightweight masonry blocks to be used for both load-bearing and non-load bearing applications.

The geopolymeric blocks were oven-dried to a constant mass and aged for 7 days at room temperature. The blocks were weighed and the dimensions were determined using a vernier calliper for accuracy.

The density was determined as follows:

$$\rho = \frac{m}{v}$$

Where:

ρ = bulk density (kg/m³)

m = mass of the specimen (kg)

v = volume of the specimen (m³)

3.8.5. Tensile Strength Test

The tensile test was performed in order to ensure material specifications to determine the quality of the geopolymer blocks. The blocks were placed in the testing machine with the required pieces and then subjected to tension until they fail (SANS 1058). The size of the specimen was 100 mm length by 100 mm width or 100 mm height. The cubes were tested for split tensile strength based on SANS 6253:2006. Mathematically, split tensile strength is expressed as the ratio of crushing load (N) to the surface area (mm²) where the load was applied. The split tensile test was applied on all specimens at 7 days of curing. Based on ASTM D1037 standards, the minimum tensile strength for portioning walls is 1.01 MPa and 2 MPA. The split tensile strength was calculated from the applied load at the point of cube failure. The average of the three tests of the cube was reported. In this study, the sample of calculation is reported in the Appendix A.

The equipment used was a King Test Auto 2000 model Pat 2001 with load capacity and load rate of 2000 kN and 40-1000 kN/min, respectively. The standards used were "Compressive Strength on concrete cubes" SANS method 5863:2006.

Based on the test, the splitting tensile strength was determined as follows:

$$S_t = \frac{2M}{\pi DL}$$

where S_t is the splitting tensile strength (MPa); M is the maximum load on the specimen (N); D is the diameter of the specimen (mm), and L is the length of the specimen (mm). Splitting tensile strength was done according to ASTM C496 and the average splitting tensile strength of three specimens was recorded. This test was conducted at the age of 7 days.

3.9. Production of non-load bearing portioning walls

The lightweight geopolymeric walls and lightweight bricks were produced by activating coal fly ash with 10 M of NaOH and adding 20% waste paper content. The mechanical properties and durability developed at this concentration during the optimization in the present study met the standard for these building materials according to SANS 204 for lightweight geopolymeric wall. The lightweight bricks were produced using 2.4 kg fly ash; 0.60 kg waste paper; 0.32 kg 10M NaOH; 0.64 kg Na_2SiO_3 and 1.8 kg water. The mixture was mixed for 45 minutes in total and cured at 80°C for 24 hours. Thereafter, the lightweight bricks were ready for application. The releasing agent used was shutter oil.

Chapter 4: Results, Analysis, and Discussions

4.1. Overview

This chapter firstly details the characterisation of coal fly ash (CFA) and geopolymer products respectively. The materials are characterised using different techniques such as X-ray diffraction (XRD) of CFA. This presents experimental results which represent the average of three consecutive measurements of geopolymers synthesized from CFA. The effect of the ratio of sodium silicate to sodium hydroxide on the compressive strength is presented, followed by the effect of the concentration of sodium hydroxide and the curing temperature on the geopolymer strength. After the mixture is developed, the effect of waste paper content on geopolymer strength, weight, shrinkage and water absorption is investigated in order to find the optimum mixture which could be applied in non-load bearing construction materials. In this section, the results of the study of the salient parameters affecting the compressive strength of LWGM and the results of the optimization of the LWGM mix composition are presented and discussed. The effects of different processing parameters on the compressive strength, water absorption and shrinkage of LWGM are discussed in section 4.3 of this section. Each of the compressive strength test data shown in figures or tables corresponds to the mean compressive strength value of the three LWGM cube block specimens tested.

4.2. Characterization of Coal Fly Ash (CFA)

In this characterisation section, CFA from Lethabo Power Station Mpumalanga in South Africa was analysed prior to geopolymer manufacturing. The CFA was characterised using various analytical techniques in order to determine the chemical composition, morphology, and the physical or crystalline structure of the material prior to its application in lightweight geopolymer development. XRD was used to determine the mineralogy of the CFA sample. XRF technique was used to determine its chemical composition.

4.2.1. X-Ray Fluorescence (XRF)

The elemental composition of Lethabo CFA used as feedstock in this study is presented in Table 4.2-1. The XRF technique was used to determine the chemical composition of the fresh Lethabo

classified CFA as described in Chapter 3. Three batches of Lethabo classified coal fly ash was analysed, named CFA 1, CFA 2, and CFA 3.

Table 4.2-1: XRF results for Lethabo Fly Ash

Major Oxides	CFA 1 (%)	CFA 2 (%)	CFA 3 (%)	Average (%)
SiO₂	56.46	56.10	56.28	56.28±0.18
Al₂O₃	29.86	30.53	30.85	30.41±0.51
Fe₂O₃	3.59	3.61	3.60	3.60±0.01
CaO	4.44	4.50	4.39	4.44±0.06
TiO₂	1.59	1.59	1.58	1.59±0.00
MgO	1.10	1.09	1.11	1.10±0.01
K₂O	0.70	0.71	0.67	0.69±0.02
P₂O₅	0.50	0.49	0.50	0.49±0.01
MnO	0.03	0.02	0.03	0.03±0.01
Cr₂O₃	0.04	0.04	0.03	0.04±0.00
Na₂O	0.41	0.27	0.35	0.34±0.07
V₂O₅	0.03	0.02	0.03	0.03±0.01
Total	99.73	100.96	102.43	101.04±1.35
SiO₂/Al₂O₃	1.89	1.84	1.82	1.85±0.36
Si/Al	2.41	2.34	2.33	2.36±0.45

XRF chemical composition analysis for CFA is summarised in Table 4.2-1. Loss on ignition (LOI) was not included to evade substantial errors caused by moisture content in samples, thus the percentages of the major elements are presented on a dry basis. Average values of major elements were recorded, and the standard deviation was quantified for errors as follows: SiO₂, Al₂O₃, Fe₂O₃, CaO, TiO₂, MgO are 56.28±0.18, 30.41±0.51, 3.60±0.01, 4.44±0.06, 1.59±0.00, and 1.10±0.01 respectively. Lethabo CFA also contains K₂O, P₂O₅, MnO, V₂O₅, Cr₂O₃, Na₂O of 0.69±0.02, 0.49±0.01, 0.03±0.01, 0.03±0.01, 0.04±0.00, 0.34±0.07 respectively.

Based on the XRF results of Lethabo station CFA, the CFA is classified as class F. The CFA was concluded to be class F as the total amount of SiO₂, Al₂O₃ and, Fe₂O₃ is 89.84% while the composition of calcium oxide is 4.44±0.06%. This observation agrees with published results on the composition of Matla CFA (Nyale et al., 2013; Madzivire, 2009; Petrik et al., 2003). The composition of CFA varies depending on the place of origin (Musyoka, 2012; Madzivire et al., 2010). Table 4.2-1 shows that the concentration of CaO and MgO is 5.54% and the total alkaline content Na₂O and K₂O is 1.03%. Silica is the most abundant compound with the mass ratio of

SiO_2 to Al_2O_3 being 1.85. The ratio of Si to Al is 2.36, based on Section 2.6. This ratio proves that class F is the ideal one for the formation of geopolymers, as observed by Wattimena et al. (2017).

4.2.2. X-ray Diffraction (XRD)

The X-ray diffraction (XRD) technique was performed to investigate the crystalline phases or the mineralogical composition in the sample of Lethabo CFA. This technique was performed as described in Chapter 3, Section 3.7.2.

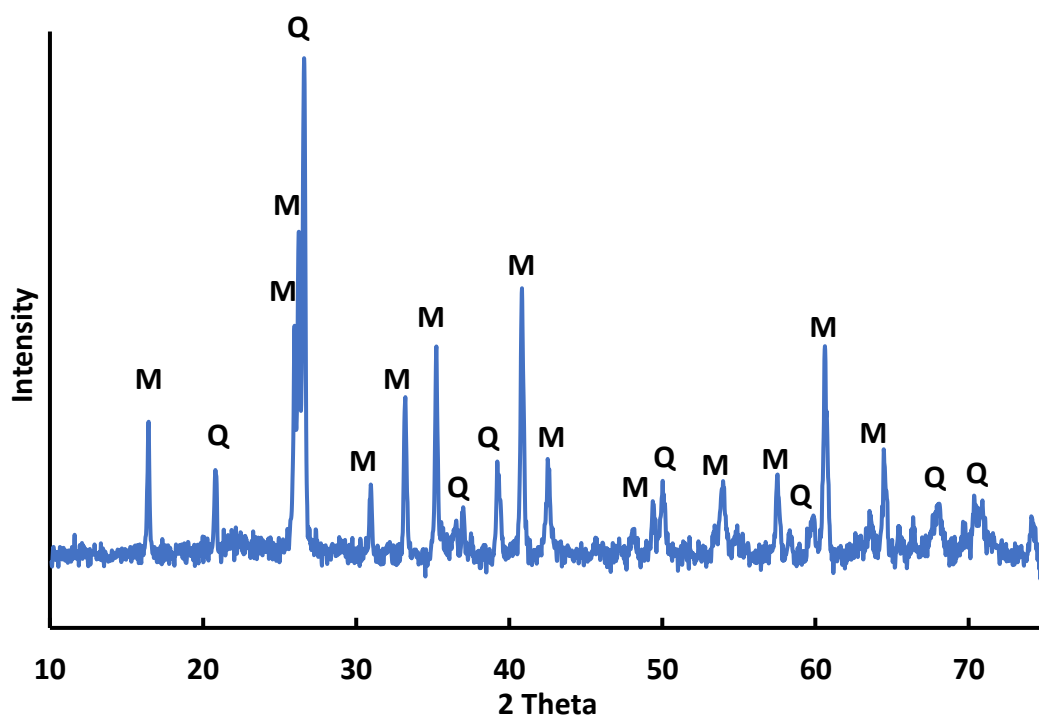


Figure 4.2-1: XRD results of CFA.

The result in Figure 4.2-2 showed that mullite and quartz were the major mineral phases present in Lethabo CFA. This observation has been confirmed by different authors (Nyale et al., 2013; Musyoka et al., 2012; Mainganye, 2012). The XRD patterns of the raw coal fly ash (CFA) have shown the presence of sharp peaks centred at 26.6° of 2θ angle due to the presence of quartz and/or mullite phase. The XRD spectrum showed a broad hump occurring between 18° θ and 30° θ which was due to the amorphous glassy phase contained in fly ash.

The mineral composition was of a low calcium Lethabo CFA which was also highly amorphous with the presence of a small amount of quartz and mullite, demonstrated by low intensity

diffraction peaks. There was, therefore, consistency between the elemental analysis obtained from XRF and the different phases obtained from XRD results, which revealed that the glass phase contained a high percentage of silica and alumina. It is always significant to know the phase composition in FA because of the fact that the amorphous glassy phase composition is one of the reactive components that can be efficiently used to form a geopolymer product.

4.3.X-ray Diffraction of Light Weight Geopolymeric Products

Figure 4.3-1 shows XRD results for geopolymers with waste paper content of 0%, 10%, 20%, and 30%. The XRD analysis procedure was described in section 3.73. The geopolymer mix contained the CFA content variation depended on waste paper content, which means that the amount of 10M NaOH and Na_2SiO_3 added was also dependant on the CFA content. 1.8kg of water was also added. The geopolymer specimen was cured at 80°C for 60 hours.

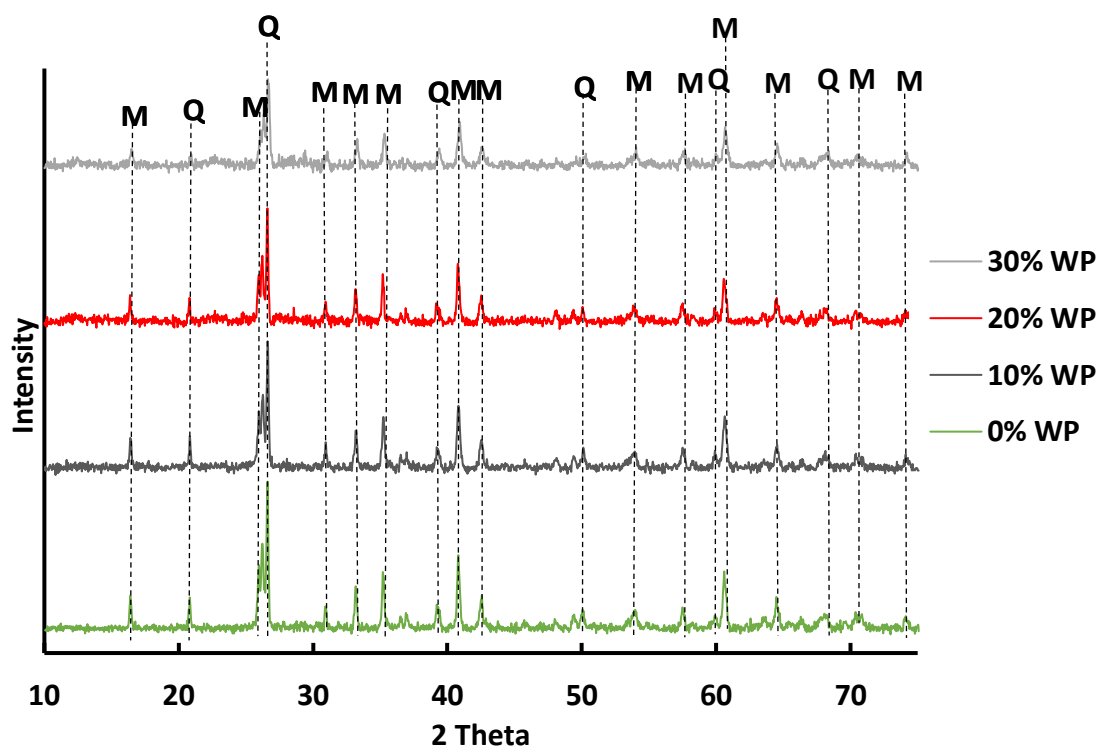


Figure 4.3-1: XRD analysis of geopolymer with different waste paper composition (0%, 10%, 20%, 30%).

The quartz/mullite phase in Figure 4.3-1 shows a reduction as wastepaper content increased and this is attributed to the dissolution of coal fly ash in alkali silicate which is a suitable condition for the nucleation of the geopolymerisation. The XRD pattern for geopolymers with waste paper content showed typical peaks of quartz and mullite. Some parts of the mullite

phase may have transformed into glassy and semi-crystalline phases (Gonzalez et al., 2003; Lukman, 2011; Lloyd et al., 2009).

4.4. Development of Lightweight Geopolymeric Material and Effect of Each Variable

An exploratory study was conducted to establish a blending method for lightweight geopolymeric materials (LWGM). Nonetheless, there is a need to optimize compressive strength in order to meet the standard requirement for lightweight non-load-bearing blocks, recommended by BS771-4:2011 to be a minimum of 1.5 MPa. Chapter 3 describes in detail the experimental procedure for the development of lightweight geopolymeric material using CFA and waste paper as principal raw material. The varied and fixed parameters were established to achieve the aims and objectives of this study. The parameters ranged from the ratio of sodium silicate to sodium hydroxide concentration, sodium hydroxide concentration, water quantity, curing temperature, rotting time and paper waste content.

The one factor at a time (OFAT) approach was adopted and used since this study focused on identifying factors that have a crucial impact on the compressive strength, tensile strength and water absorption of LWGM and not on the interaction between the factors (Montgomery, 2013). The OFAT approach is a standard form of experimental design that requires the selection of a baseline starting point for each factor or (baseline set of levels for each factor) followed by a successive variation of each factor over its scope with the other factors held constant at the baseline level (Montgomery, 2013). The effect of the abovementioned parameters was studied as described below.

4.4.1. The Effect of Na_2SiO_3 to Compressive Strength

The procedure used in this section was described in Chapter 3. This section involved the study of the influence sodium silicate had in the geopolymerisation process and its effect on the compressive strength of the dried geopolymer. The aim of the research is to develop high strength, cost-effective material; minimising the use of chemicals and materials is one way of reducing the costs. Therefore, the effect of sodium silicate was investigated with the aim of removing it as part of lightweight geopolymeric materials. This investigation investigated

whether to use sodium silicate or just use sodium hydroxide alone. The effects of sodium silicate were explained in Chapter 2, Section 2.5.2.

Six levels (0, 0.5, 1, 1.5, 2, and 2.5) of sodium silicate to sodium hydroxide ratio were utilised in this investigation. In Figure 4.3-1, these amounts of sodium silicate and sodium hydroxide were mixed separately with 3 kg of CFA and 0 kg of water. This study was done without the addition of waste paper content to analyse the effect of sodium silicate on geopolymer strength. Because waste paper that absorbs water was not added to the formulation, the water in the system was enough for adequate workability. The concentration of NaOH used was 12M. The mixing time was kept constant at 30 minutes. The paste was cast in 100 mm³ moulds and covered with plastic to avoid water/moisture loss. The temperature used for curing was 80 degrees celsius for 24 hours, and dry samples were stored at room temperature for 7 days, as described in Chapter 3.

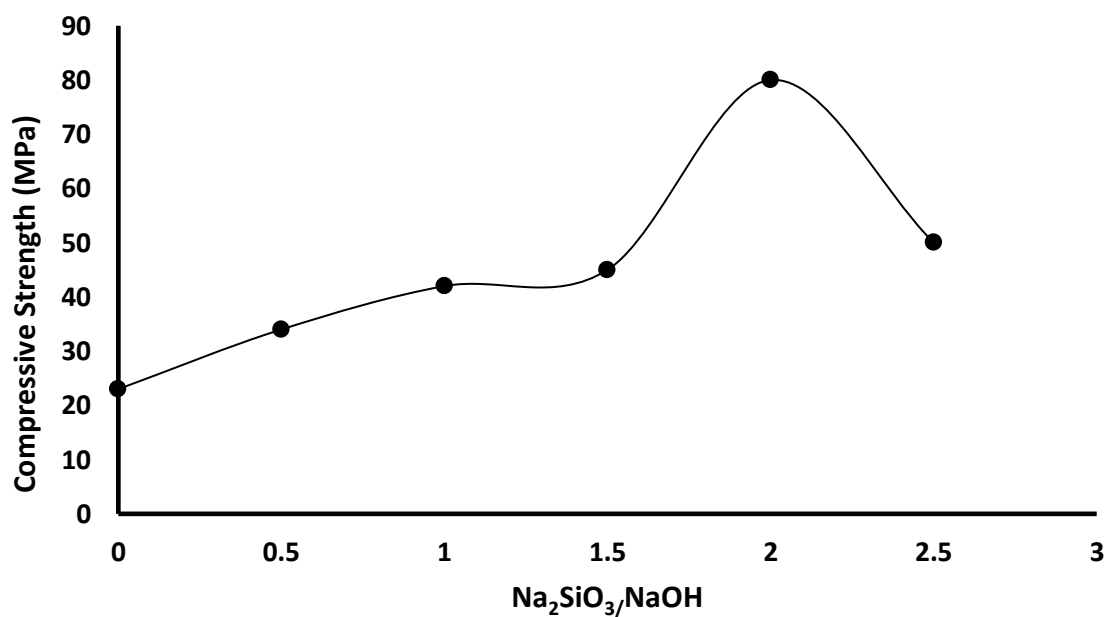


Figure 4.4-1: The effect of SS/SH ratio on compressive strength.

Figure 4.4-1 shows that the compressive strength is the highest when the ratio of Na₂SiO₃/NaOH (S/N) is 2.0. As the Na₂SiO₃/NaOH ratio increases from 2 to 2.5, the compressive strength decreases. Whereas there is a significant increase in the compressive strength when the ratio increases from 0 to 2.0 (80 MPa). The process of geopolymerization is essentially enabled by the initial curing at an elevated temperature ($\pm 80^{\circ}\text{C}$) for 24 hours. In addition, with a mass ratio of 2:1 of Na₂SiO₃/NaOH, the dissolution of silica and alumina was strong, leading to an increase in the compressive strength of the ash geopolymer.

These results demonstrated that the compressive strength of the geopolymer products increased linearly as the amount of sodium silicate increased up to a ratio of 2:1. Therefore, the optimum ratio of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ in the synthesis of lightweight geopolymeric material was determined to be 2 in this study. Deducing from the results in Figure 4.4-1, it can be said the compressive strength increases with an increase in sodium silicate. However, as the ratio increased to 2.5 there was a drop in compressive strength, and this can be attributed to an excess of sodium silicate that interfered with the geopolymerisation process. It can be further explained by describing the role played by sodium silicate as previously discussed in Chapter 2 section 2.5.2. When using solid silicate, the silicate species must first be removed from the solid origin through alkali and water reactions leading to a more gradual release of silica to the geopolymer gel. This may potentially give greater control over the properties of the gel by regulating the speed of release of silicate, which can be controlled by altering the solubility of silicate. Essentially, the increase in the $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio resulted in an increase in the sodium content of the mixture. Sodium is essential for the formation of geopolymers as it acts as a load-balancing ion. Nevertheless, the compressive strength decreases as more silicate is applied to the process, as excess sodium silicate prevents water evaporation and structural formation (Morsy et al., 2014).

The objective of this particular study was to investigate if it is necessary to totally eliminate Na_2SiO_3 from the main study. As the results showed, the role played by Na_2SiO_3 in the geopolymerisation process is quite significant as it balances out the ions involved during the formation of geopolymer gel. At 0 sodium silicate, the strength was 23 MPa, which is low. Furthermore, the results obtained during the study of the effect sodium silicate showed that it is important to add sodium silicate to the system. NaOH is responsible for the activation of the geopolymer formation process as, during the dissolution process, the high concentration of OH^- from NaOH in the system is responsible for the hydrolysis of the silica and alumina bonds from the aluminosilicate. Therefore, the effect of the NaOH concentration was studied in the following section.

4.4.2. The Effect of NaOH Concentration on Compressive Strength

In this section, the optimum ratio of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ used was 2 and in mass bases, it was 1.2 kg Na_2SiO_3 mixed with 3 kg of FA, 0.4 kg of NaOH solution of different molarities (10M, 12M and 14M) cured at 60°C and 80 °C for 24 hours and aging time of 7 days at room temperature

(as described in Chapter 3, Section 3.3.1). These conditions were used to investigate the influence that NaOH molar concentrations have on the development of geopolymer compressive strengths.

In the present study, the concentrations of NaOH varied were 10, 12 and 14M for each concentration of NaOH, while 0.4 kg was the mass used of the liquid solution. The compressive strengths of geopolymer materials developed using these concentrations of NaOH are depicted in Figure 4.4-2.

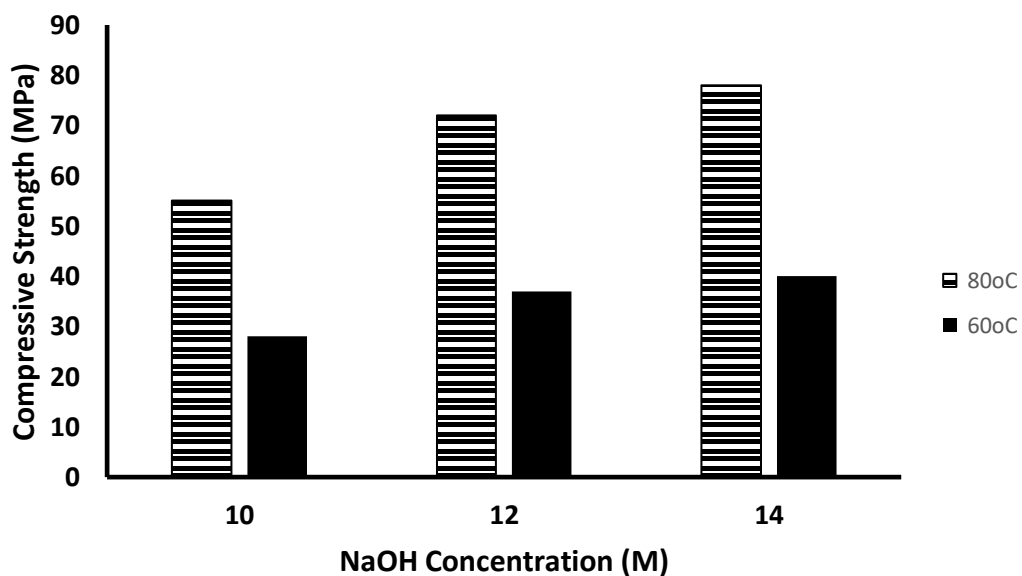


Figure 4.4-2: Effect of NaOH concentration on compressive strength.

In Figure 4.4-2, the compressive strengths of the geopolymer materials obtained were 55 ± 1.5 , 72 ± 4.5 and 78 ± 3.5 , when activated with 10, 12 and 14 M respectively at 80 degrees Celsius. These results have shown an incremental value of about 10 MPa as the concentration of NaOH increased with 2 M (from 10 to 14 M).

Figure 4.4-2 showed that the highest compressive strength was 78 ± 3.5 MPa obtained when CFA was activated with 14 M of NaOH using the conditions described in this section. It was noticed that 10 M of NaOH developed only a low compressive strength, which could be attributed to the low dissolution of silicon and aluminium ions in minimum sodium hydroxide solution. Chemically, the use of a low alkali solution produced a weak hydrolysis reaction. However, when CFA was activated with 14 M of NaOH, the compressive strength did not improve by a huge percentage (78 ± 3.5 MPa). This could be attributed to the fact that the concentration of ion hydroxide was high enough, the dissolution of CFA was increased, but

polycondensation was held back. Although the increase in alkali concentration increased the intensity of the geopolymers from 10 to 12 M NaOH, as shown in Figure 4.4-2, the excess concentration of hydroxide ion may cause aluminosilicate gel precipitation (Somna et al., 2011; Zuhua et al., 2009) at a very early stage. The result was a lower strength geopolymer, which was the case for the geopolymer triggered with 14 M of NaOH.

Abdullah et al. (2011) developed geopolymer material with a compressive strength of 69 MPa using 12 M of NaOH and this was their optimum concentration; the conditions used were the ratio of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ of 2.5, ratio of CFA/alkaline activator of 2.5, cured at 70°C for 24 hours and aged for 7 days. In Abdullah's studies, the curing temperature was higher, and a low amount of CFA and a large volume of alkaline activator was used, which could be the reason for their finding that 12 M of NaOH was the optimum activator which could surely have an impact on the cost of geopolymer production.

Singh et al. (2016) reported that the highest compressive strength of 68 MPa was obtained when CFA was activated with 14 M of NaOH using a 2.5 ratio of $\text{Na}_2\text{SiO}_3/\text{NaOH}$, cured at 60°C for 24 hours and aged for 7 days; however, Singh et al. added fine sand and aggregates which constituted about 77 % by mass of all mixture, and naphthalene-based high-range water-reducing admixture was also added. Thus, not only was the compressive strength lower in the literature (Singh et al., 2016) than what was developed in the present study but also their process was very expensive due to the additives and a low amount of coal fly ash that was used.

In the present study, the liquid ratio by mass of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ was 2 and the ratio by mass of CFA/alkaline activator was 2.5. In this section, the highest compressive strength (72 ± 2 MPa) of the geopolymer obtained was activated with 14 M of NaOH. The objective was to use fewer chemicals whilst maintaining the properties in order to reduce the cost of the product as depicted by figure 4.3-2. 12M and 14M have a slight difference in strength, therefore based on cost-effectiveness 12 M uses less sodium hydroxide yet provides almost same compressive strength as 14M. The minimum strength required for non-load bearing materials ranges from 1.5 MPa to at least 3 MPa, therefore 10 M NaOH concentration is sufficient for lightweight geopolymeric material as the compressive strength obtained using 10M is 55 MPa. Therefore 10 M NaOH concentration was used for the development of lightweight geopolymeric materials

4.4.3. The Effect of Curing Temperature on Compressive Strength

The optimum parameters obtained in section 4.4.1 and 4.4.2 were used in this section. Thus, the investigation of the effect of the curing temperature was performed using the fixed parameters constituted of 0.4 kg of NaOH solution (10 M), 0.8 kg of Na₂SiO₃, 3 kg of FA and aging time of 7 days at room temperature as described in Chapter 3 (section 3.3.1). The varied curing temperatures during the geopolymerisation were 60 and 80°C for 24 hours. In this section, the average of the results obtained in triplicate has been depicted in Table 4.3-1.

Table 4.4-1: Effect of Temperature on compressive strength

Temperature °C	Comp Strength MPa
80	55.22+/- 0.3
60	42.3+/- 0.4

The effect of temperature with respect to the compressive strength is shown in Table 4.3-1. These results demonstrate that the increase in temperature increased the compressive strength of the geopolymer. The temperatures used and the compressive strengths obtained were 60 and 80°C, and 42.3 ± 0.4 and 55.2± 0.3 MPa, respectively. The highest compressive strength (55.2± 3 MPa) was observed using the temperature of 80 °C; there was a slight improvement in terms of the compressive strengths developed by the curing temperature of 80° C. This showed that the increase in temperature accelerated the alkali activation of fly ash.

Based on Davidovits (1991), 100° C and below is the temperature needed for the crystallization of geopolymer materials. Other researchers have proposed a curing temperature of 80°C for the processing of geopolymers using class F CFA (Morsy et al., 2014; Bakkali et al., 2016; Kamarudin et al., 2011). Furthermore, Rangan et al. (1987) reported that the highest compressive strength of 70 MPa was observed after curing the geopolymer samples at 80°C for 48 hours, an aging time of 7 days, with aggregates added to their mixtures. Not only was the compressive strength reported by Rangan et al. (1987) lower than what is reported at 80°C in this study but also, they added aggregates to their mixture and cured the geopolymer for 48 hours at 80°C whereas in the present study no aggregates were used, and the curing time was only 24 hours at 80°C.

Approximately similar experimental conditions used in the present study were applied by Abdullah et al. (2011). However, their results showed that 60°C was the best curing temperature

to obtain a compressive strength of 71.04 MPa, and a compressive strength of 30 MPa was obtained at 80°C; this could be attributed to the different conditions and formulations used in the geopolymerisation process, which has an effect on the settling and strength development of the geopolymer (Wattimena et al., 2017). It is also due to a significant loss of moisture from the sample as the strength deteriorates when moisture evaporation is allowed (Bakharev, 2005). It is therefore proposed that geopolymer reactions require the presence of moisture to develop good strength (Chindaprasirt et al., 2007). The compressive strength developed in this section was higher than reported in the literature and was different to many of the previous authors, such as Abdullah et al. (2011), and Hardjito et al. (2004), regarding their experimental procedures, developed formulations, the results they obtained and conclusion they made.

4.4.4. The Effect of Compacting Forces on the Compressive Strength

The aim of this section is to investigate the effect of compacting pressure on compressive strength. In masonry blocks, the rise in moulding pressure increases the compressive strength (Riza et al., 2011; Bahar et al., 2004) and the binding material becomes more active at higher moulding pressure compared to lower moulding pressure. In order to investigate the effect of the compacting pressure on the compressive strength of the LWGM, the compacting pressure ranged from 1.25 MPa to 7.5 MPa while the other parameters remained constant: 10 M NaOH, Na₂SiO₃/NaOH ratio of 2 and 80°C for curing. 40%, 30%, 20% and 10% WP content were investigated.

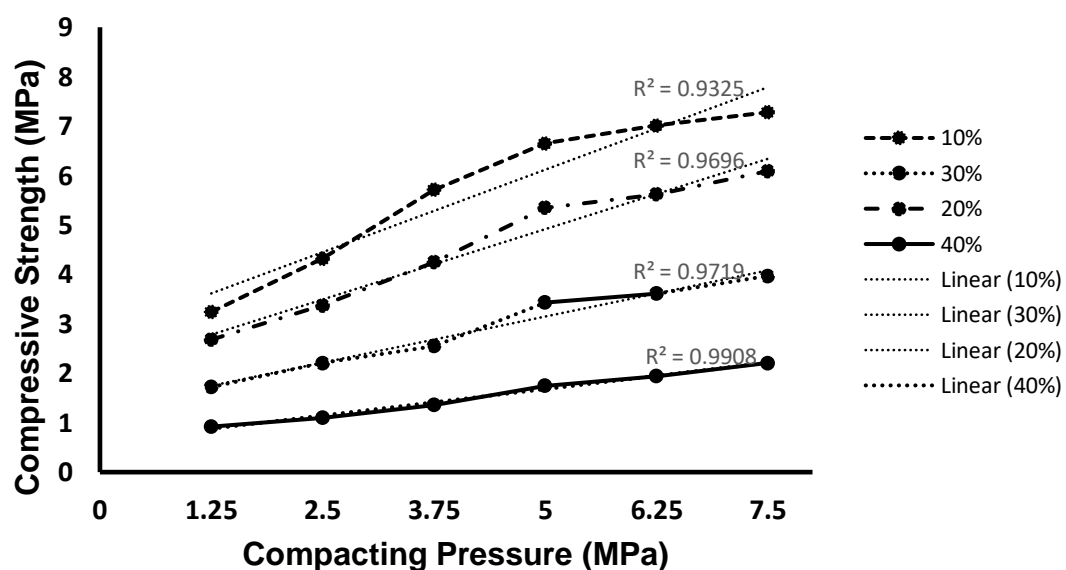


Figure 4.4-3: The effect of compacting pressure on compressive strength.

As shown in Figure 4.4-3, the compressive strength of LWGM increases as the compacting forces increase. The compressive force decreases with the change in the compacting force applied. The effect of pore filling, homogeneity and enhanced bonding at high compacting pressure can be the reason for high compressive strength observed within the block microstructure. According to literature, the compacted block samples exhibit air spaces and low density at low moulding pressure, while at higher moulding pressures they display reduced voids, higher compaction and density (Subramania & Benny 2013). The optimum compacting pressure chosen from the experiment is 5 MPa as from 6.25 MPa there is no significant increase in compressive strength, and this can be attributed to the liquid solution which was collected from the geopolymer as it was being compressed. The liquid solution has water and alkali activator which are important for geopolymerisation and responsible for strength enhancement. This result showed the need to regard the compacting force as part of the LWGM optimization function in order to achieve optimum processing parameters. In addition, the fitted regression line shows that the R² values for each waste paper content/composition range from 0.93 to 0.99, which suggests that there is a strong relationship between the compressive strength and the applied compacting stress. Based on the study it is noted that 5MPa is the compacting pressure that will be used in the next session where the effect of waste paper content on compressive strength will be investigated.

4.4.5. The Effect of Waste Paper Content on Compressive Strength

Based on the optimum conditions obtained from section 4.4.1 to 4.4.5, Na₂SiO₃ to NaOH ratio of 2, NaOH concentration 10 M, FA weight varied with waste paper content and curing temperature of 80°C. The geopolymer was cured at 80°C for 60 hours to allow the geopolymer to dry as pulp had a large amount of water. The compaction pressure used was 5 MPa in this section. This section investigated the effect of waste paper content on geopolymer strength as this is the purpose of the study.

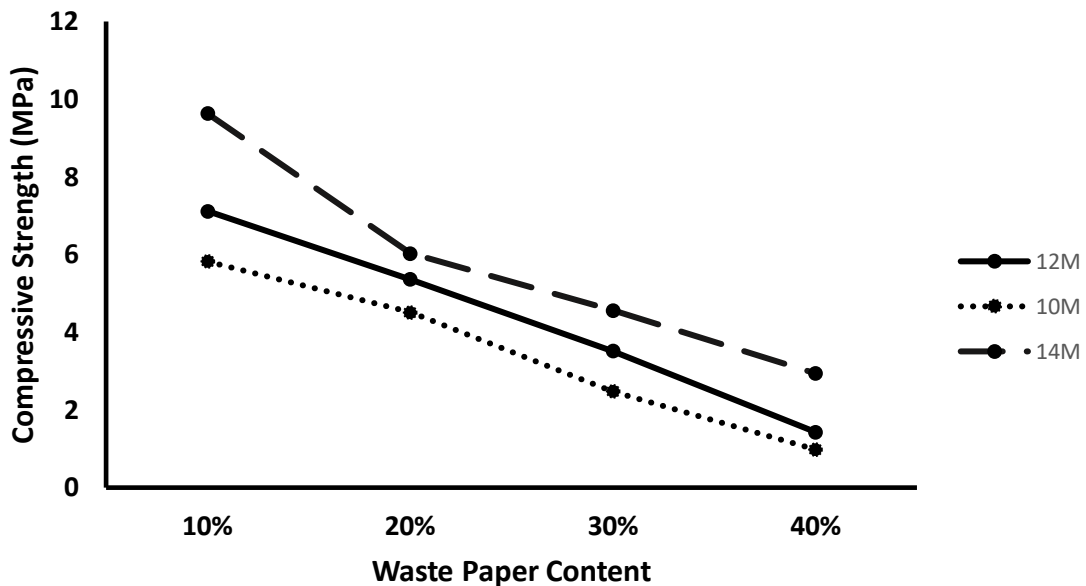


Figure 4.4-4: Effect of WP content on compressive strength using 12M NaOH concentration and 80°C.

As figure 4.4-4 depicts, when 10M NaOH concentration is used, 10 % WP content is 5.82 MPa, 20% WP content is 4.51 MPa, 30% WP is 2.48 MPa and 40% is 0.98 MPa. At all NaOH concentrations, the phenomenon is related to the rise in the volume of waste paper, which reduces the compressive strength. Based on the graph in Figure 4.4-4, it can be inferred that as the content of waste paper increases, the compressive strength decreases. The trend observed in Figure 4.4-4 can be attributed to the paper properties; although the paper is known for its lightweight properties, it does not have desirable strength properties.

At high waste paper content, the geopolymer becomes brittle and this can be attributed to the voids in the specimen which are caused by increasing cellulose fibre pulp which is low strength in the geopolymer. Therefore, this leads to low strength as well as low density. There is poor bonding between cellulose particles and geopolymer particles which increases tiny voids. The required standard of ASTM C129 for non-load bearing materials is more than 1.5 and based on the results of this study, 20% and 30% pass the required standards of ASTM. For SANS, only 20% waste paper content passes the requirement, as it is 3.5 MPa for non-load bearing materials

Observing literature on studies done for the synthesis of waste paper reinforced geopolymers Anuara (2015) investigated the effect of incinerated waste paper on geopolymer strength. Waste-paper sludge ash and a mixture of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) were used as binders. Anuara's studies showed the optimum mix to have 12M NaOH

with the highest compressive strength of 6.788 MPa. Momin & Sayyad (2017) investigated papercrete brick and the optimized mix was 1:1.5:4:2 (cement: fly ash: sand: paper) resulting in a compressive strength of 3.24 MPa that is higher than traditional clay bricks and meets the IS code recommendation. To conclude, Pachamutu & Thangaraju (2017) investigated the development of a CFA and the incineration of paper sludge as ash as source material for the preparation of geopolymer concrete. The specimens were prepared with varying percentages (0 %-20 %) of CFA replaced by paper sludge under different curing regimens. At 20%, the maximum strength was 20 MPa.

As other studies have shown, Anuara (2015) produced 6.8 MPa, Momin & Sayyad (2017) produced 3.24 and Pachamutu & Thangaraju (2017) produced quite a higher compressive strength of 20 MPa and 20% waste paper. These studies did not use fly ash and waste paper; they all had aggregates such as cement and sand. However, for the current study, waste paper was not incinerated as this would have consumed energy as the process requires an oven to be used at higher temperatures, namely 120°C to 150°C for as long as 80 hours. This current study has waste paper as the only aggregate of the experiments and managed to obtain above minimal required strength by ASTM 1.5 MPa and SANS 2.8 MPa. This current study has waste paper as the only aggregate of the experiments and still managed to obtain above minimal required strength by BS, ASTM, and SANS. Based on the current study it can safely be concluded that CFA and waste paper (no incineration) are able to produce high strength geopolymeric material without extra aggregates.

4.4.6. The Effect of Water Content on Compressive Strength

Water is important in the manufacture of bricks and concrete as it reacts chemically with cement to create a paste that attaches to aggregates. However, for geopolymerisation, as noted by Sathonsaowaphak et al. (2009), the addition of water to the geopolymerisation process enhances workability in order to make it easier for moulding. The current study concludes that it is therefore important to add just sufficient water to geopolymerisation. Paper is known for its properties of absorbing water and waste paper was one of the important raw materials water was used for the synthesis of LWGM in this study. Waste paper was provided as pulp with 70-75% water content.

Chapter 3 noted the procedure whereby the pulp was added after CFA and alkali activator liquids were mixed. By adding waste paper the paste increased in viscosity which resulted in moulding being difficult, hence the hydraulic press compacting method was proposed. In order to investigate the influence of the water content on the compressive strength of the mixture, different sets of mixtures containing differing proportions of the water content by weight of waste paper were made. The ratio of waste paper to water used in the experiments were 0.3, 0.6, 0.9 and 1.2 respectively. Originally the ratio of water in pulp from the factory was 0.6. In this section, water was added to the paste to improve workability to study the effect of water content on geopolymer strength and shrinkage.

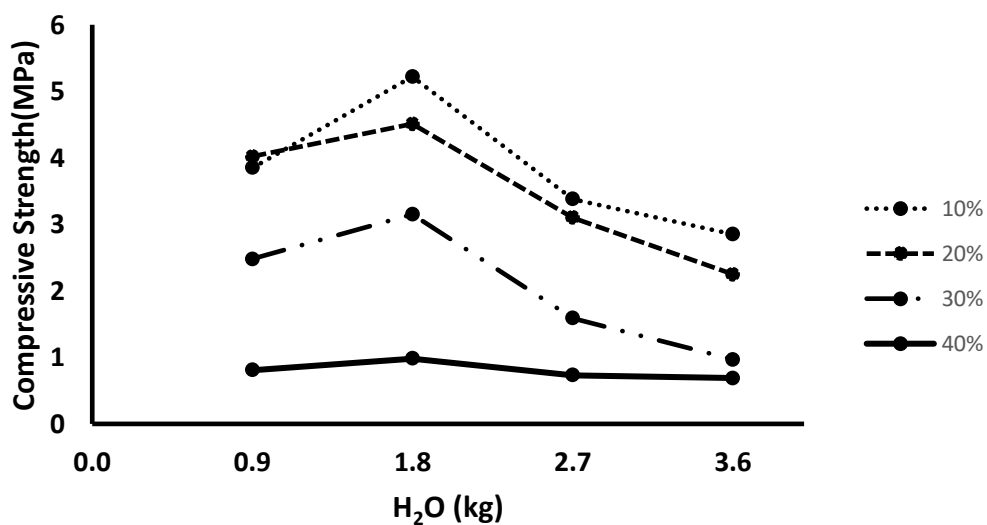


Figure 4.4-5: Effect of water on compressive strength.

Figure 4.4-5 presents the effect of water content on compressive strength. The graph indicates that as the water to waste paper ratio increased the compressive strength increased. The compressive strength increased with less water in the geopolymer synthesis process. A decrease in water increases sodium hydroxide concentration in the aqueous phase (Patra et al., 2011) which enables the activation of alumina and CFA. At 1.8 kg of water content, the compressive strength increased compared to 0.90 kg, 2.70 kg and 3.60 kg. The geopolymerisation process requires water, as discussed in Chapter 2, Section 2.6.1. At 0.90 kg the compressive strength was low because there was not enough water to mix the CFA with alkali activator and waste paper. This resulted in a paste with less workability and difficulty in moulding, which left voids that negatively affected the compressive strength. At 2.70 kg and 3.6 kg the water was more than sufficient, which affected the geopolymerisation process as discussed in Chapter 2, Section 2.6.1. Therefore, it is preferable to decrease the amount of water

added in the geopolymerisation process as water affects the geopolymer's compressive strength.

According to Oyirom (2016), waste paper is a fibrous material, therefore it is advisable to use a compacting method other than vibration. Oyirom conducted a study comparing hydraulic press and tamping rod effect on the development of cementless lightweight construction material, and she concluded that the less amount of water involved, the higher the strength. Further, the hydraulic press was the compaction method of choice as it is less affected by human error and the pressure used to compress the mould will be constant. In Figure 4.3-5 the trend shows that as the water content increased, the compressive strength increased linearly up to 1.80 kg. However, from 1.80 kg there is a decrease in compressive strength, and this can be attributed to the low water content in the geopolymer.

During the geopolymerisation process it is necessary to have sufficient water content, otherwise coal fly ash will not be completely wet, considering that waste paper is also being added, and paper is known for its ability to absorb water. This will have an impact on the workability of the paste, making it difficult to mould the samples. This difficulty in moulding results in voids which will reduce the mechanical properties of the geopolymer. Therefore, in this study 1.8kg of water which is a ratio of 0.6 of water to solids (CFA and WP) is the optimum water content for the geopolymerisation mix.

The results obtained in Figure 4.4-5 agree with the observation reported by Hardjito et al. (2004) and Patankar et al. (2013). These researchers also observed that the flow of the geopolymer increased with an increase in the water to the geopolymer ratio, while the compressive strength of the geopolymer decreased with an increase in the water to the geopolymer binder ratio. In this section, the amount of water was proven to be very significant and effective for the development of the strengths of the final geopolymers.

Below is a figure showing the effect of water on geopolymer shrinkage.

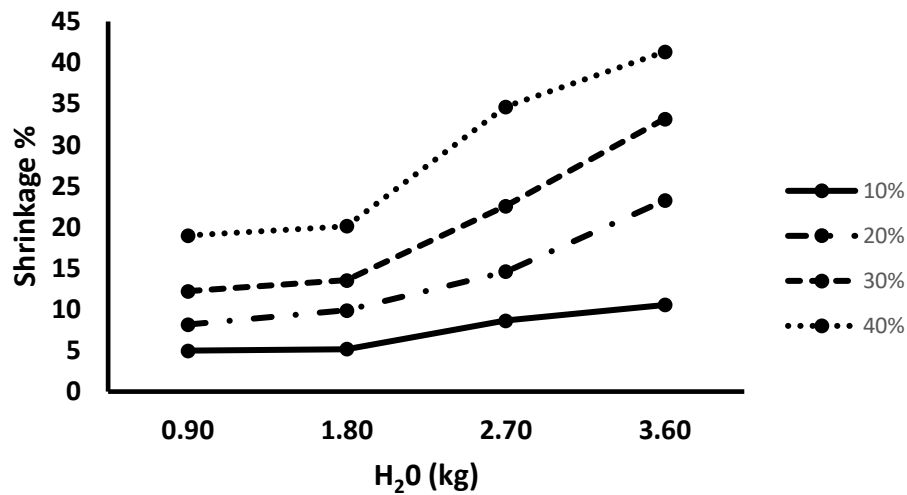


Figure 4.4-6: Effect of water on geopolymer shrinkage.

After studying the effect of water content on compressive strength the effect of water content on geopolymer shrinkage was studied, as shown in figure 4.4-6. As Figure 4.4-6 depicts, as the water increased to 1.80kg the more the geopolymer shrank during the curing stage. The geopolymer shrank due to the amount of water loss. This material shrinkage was inadvisable as it resulted in a geopolymer losing the shape as the material imploded during curing. Based on both Figure 4.4-5 and 4.4-6, less water is advisable; the optimum is 1.80 kg, which is water that is already in the pulp at 20% waste paper content. Therefore, there is no need to add more water to the system as the water in the pulp does not cause a huge percentage of shrinkage.

As the water content increased, the shrinkage percentage increased. This trend can be attributed to waste paper in the geopolymer. Waste paper absorbs a lot of water and cellulose contains linear strands that are attracted to each other by hydrogen bonding and twist to make fibres. Water could break these intermolecular attractions and substitute intra-molecular attractions upon drying, causing each polymer molecule to take up less space. Paper is usually rolled and dried under pressure of rollers. The dried paper is essentially stretched, leaving residual strain in it. Wetting the paper allows it to contract when it dries again, without the stretching force being present. This combined with molecular folding rearrangement could also cause wrinkling of the sheet giving apparently greater shrinkage. The relationship between shrinkage and compressive strength is displayed in figure 4.4-7; the R squared is 0.89 which shows that there is a relationship between compressive strength and shrinkage. As shown in figure 4.4-7 the compressive strength decreases with an increase in shrinkage.

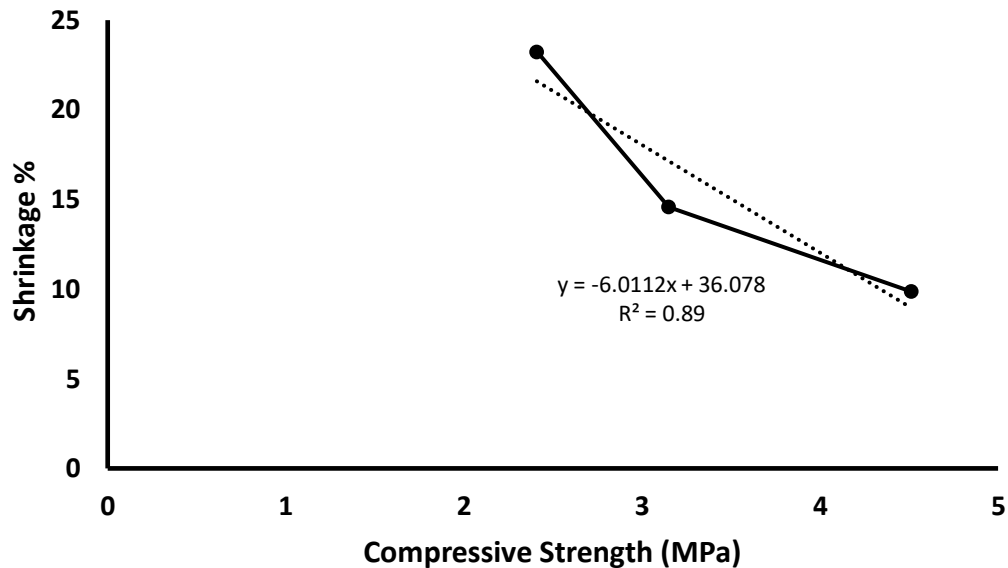


Figure 4.4-7: Relationship between compressive strength and shrinkage

4.4.7. The Effect of Waste Paper Content on Geopolymer Weight

The aim of this study is to synthesize lightweight geopolymer material used for non-load bearing construction materials; therefore, the focus of this section is to develop a lightweight geopolymeric material by varying waste paper content from 0% to 40%. Based on the optimum conditions obtained from section 4.4.1 to 4.4.6, Na_2SiO_3 to NaOH ratio of 2, NaOH concentration 10 M, CFA content varied based on waste paper content, as shown in chapter 3, at curing temperature 80°C for 60 hours with 1.8 kg of water or 0.6 ratios of water to solids. As described in Chapter 3, the procedure for making lightweight geopolymers using waste paper as the aggregate is similar to making ordinary geopolymer except waste paper is then added to the geopolymer paste and further mixing is attained. As waste paper is a fibrous and voluminous material the compacting method used was hydraulic press, and 5 MPa were applied in this section. As Figure 4.4-8 depicts, the geopolymer weight reduced as waste paper content increased. The aim of this study was to develop a lightweight geopolymer and as the graph indicates, the weight significantly decreased. The reference geopolymer from the study had an average weight of 1.72 kg which makes 20% waste paper content more than 50% lighter with 0.82 kg. The construction industry has shown interest in waste paper due to its desirable strength to weight ratio and low density.

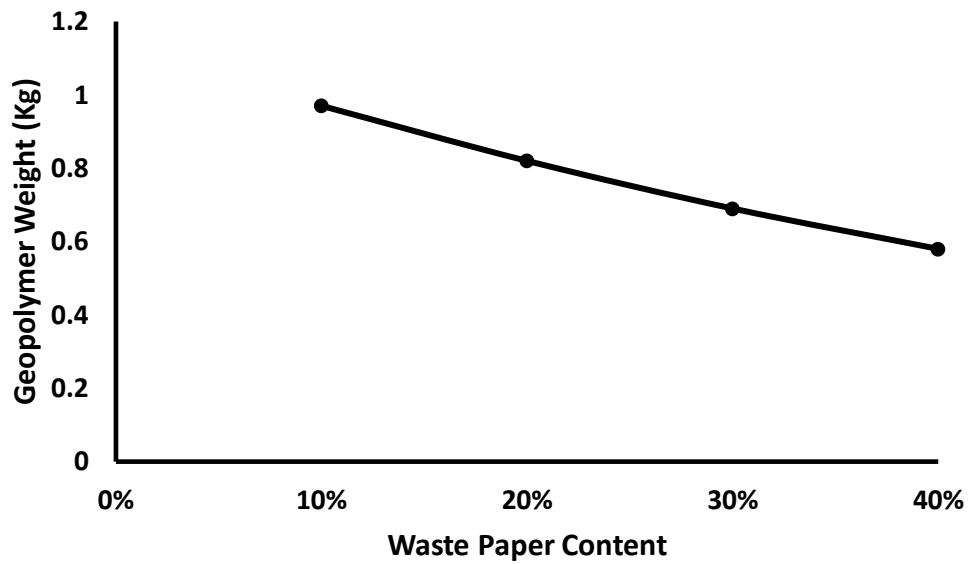


Figure 4.4-8: Effect WP on geopolymer weight using NaOH 12M and 80°C

Figure 4.4-8 depicts the effect of WP content on geopolymer weight. Referring to the graph above as waste paper content increased the geopolymer weight decreased. The results shown in figure 4.4-8 are satisfactory as the use of waste paper was to have a lightweight geopolymer.

Figure 4.4-9 shows the effect of waste paper on density.

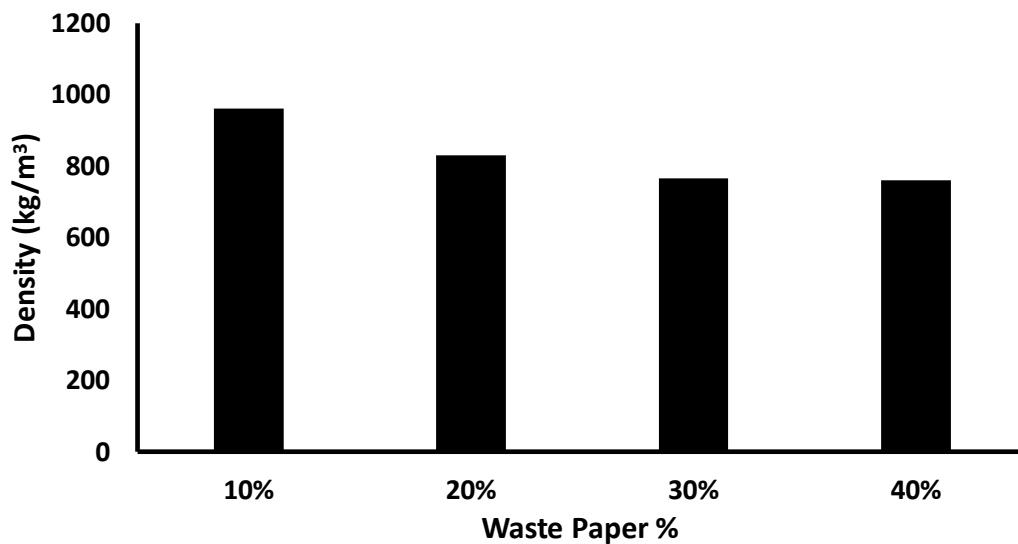


Figure 4.4-9: The effect of waste paper content on geopolymer density.

Figure 4.4-9 represents the effect of waste paper content in geopolymer density. The geopolymer density decreased with an increase in waste paper content. 10%, 20%, 30% and 40% waste paper content had the density of 929.9, kg/m³, 829,5 kg/m³, 766.2 kg/m³ and 760.5 kg/m³ respectively. The apparent density of lightweight material used for non-load bearing wall ranges between 500 kg/m³ to 1500 kg/m³ (BS EN 771-4:2011). The results shown in Figure 4.4-9 are in the range of lightweight construction materials. The density displayed in figure 4.4-9 satisfied the density range of 300-1000 kg/m³ (BS EN 771-4:2011) and 625 kg/m³-1500 kg/m³ (BS EN 2028 1975) specified for lightweight non-load bearing blocks.

The densities shown by the geopolymer sample are comparatively lower than the average density of 1060,74 kg/m³ recorded for the papercrete block produced by Akinwumi et al. (2014). The use of lightweight blocks in building is capable of increasing productivity as well as reducing construction time. The less labour-intensive characteristics associated with the use of lighter blocks allow workers to be more effective during construction.

Figure 4.4-10 shows the relation of LWGM density with compressive strength. It is evident from the graph that the compressive strength is proportional to the density of the specimen. The R² value for the fitted polynomial trend line was found to be 0.9479 which indicates the existence of a relatively strong correlation.

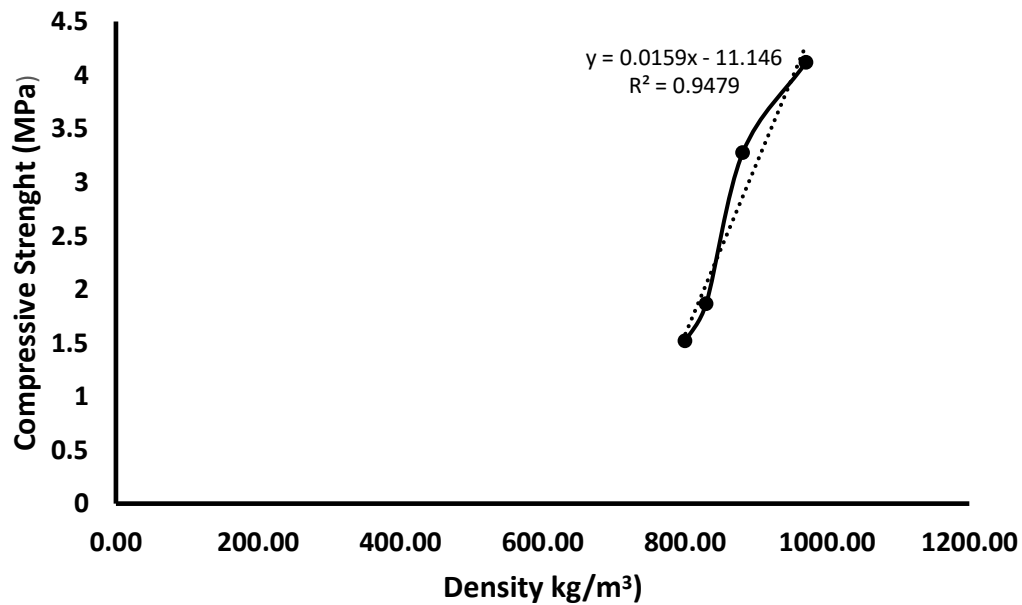


Figure 4.4-10: Relationship between density and compressive strength.

In figure 4.4-10 the compressive strength decreases with a decrease in density. This can be attributed to the paper characteristics, as waste paper content increases voids in the specimen, which makes the geopolymer lighter and the voids weaken the specimen, resulting in low compressive strength.

4.5. Durability Test

4.5.1. Effect of Waste Paper Content on Tensile Strength

This section involved the study of the impact that the amount of waste paper had on geopolymer split tensile strength. There were four levels of waste paper content that were tested, 10%, 20%, 30%, and 40%. The results of splitting tensile strength of the lightweight geopolymer material at 7 days aging are given in Figure 4.5-1 below.

Figure 4.5-1 shows the tensile strength obtained for each waste paper content. It is observed that there is an increase when waste paper content increases from 10% waste paper content to 20% and there is a decrease in split tensile strength from 30%. Further increase of waste paper content, however, led to a decrease in the tensile strength. The decrease in tensile strength can be attributed to the bonding of waste paper fibre strands and geopolymer molecules, and an excess amount of paper hinders the geopolymerisation process which will result in tiny voids that create weak geopolymer. Mansur and Aziz (2013) have reported a

similar pattern for jute fibre reinforced cement paste. The improved ductility comes from the debonding and pulling out of the fibres that connect the cracks.

Comparing the 20 % waste paper content to 10 % waste paper content geopolymer, the split tensile strength is slightly improved because the coexistence of the secondary calcium silicate hydrate phase with the primary geopolymer phase is another reason for improving the mechanical strength, as observed by Yip et al. (2005). But as waste paper content increased further, the intensity of the split tensile strength decreased. Jing Liu et al. (2014), while using palm oil ash as a binder for Geopolymer concrete, reported similar findings.

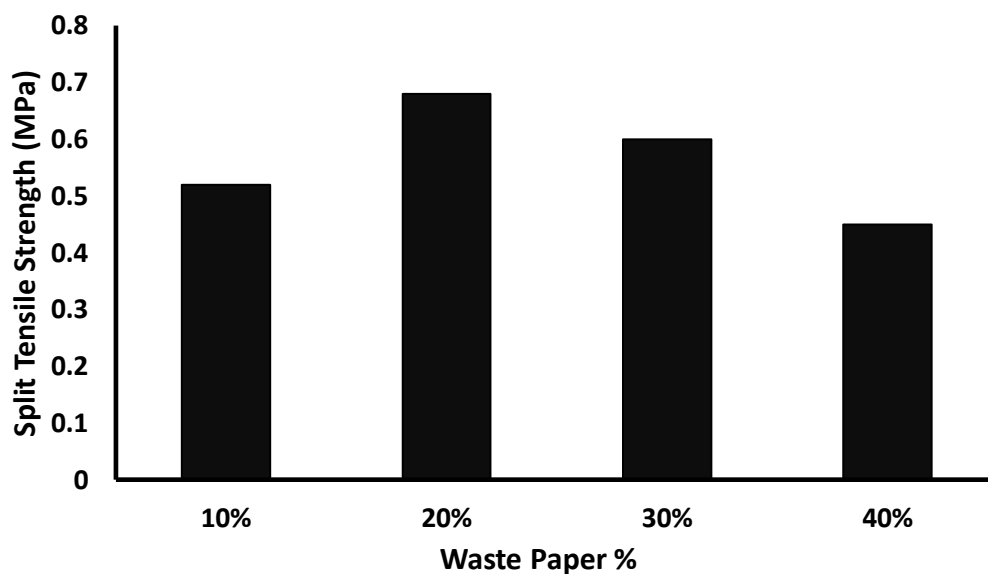


Figure 4.5-1: The effect of waste paper content on split tensile strength.

The analogy is limited to CFA-based on geopolymer (paste, mortar, and concrete), with and without the reinforcement of fibres. The CFA-based geopolymer has to break tensile strength values, with most of the values varying from 1 to 5 MPa, between approximately 0.25 and 7.5 MPa. ASTM D1037 requires a minimum tensile strength of 0.83 and 1.02 MPa for a perpendicular wall. Figure 4.4-1 shows that 20 % meets the minimum requirements for the tensile strength of drywall/non-load bearing walls.

4.5.2. Effect of Waste Paper Content on Water Absorption

It is necessary to know the durability properties of a building material, such as water absorption, fire resistance, and thermal conductivity for the material to function effectively during its service life. In this section, water absorption of the lightweight geopolymeric material will be

studied as the LWGM produced in this study is not expected to be exposed to the weather elements as it is intended to be used for non-load carrying purposes. Nevertheless, as a novel building material, finding its sorptivity level was crucial in order to determine its water absorption ability with the aim of making suitable recommendations for its installation.

Knowledge of indoor building or outdoor building water absorption is critical for a building material to function effectively during its service life as it allows the determination of its hydrometric properties. Karagiannis et al. (2016) argued that by providing adequate preventive measures at the design stage, moisture-related problems in the building can be effectively balanced. In the case of a new building material, however, it is necessary to determine its water absorption rate in order to determine the appropriate recommendation for its installation.

Figure 4.5-2 shows the amount of water absorbed per each 0.001 m³ cube cured at 80°C and aged for 7 days. 2.4kg of CFA, 0.6 kg of WP, 0.32 kg of 10M NaOH, 0.64 kg of Na₂SiO₃ and 1.8 kg H₂O.

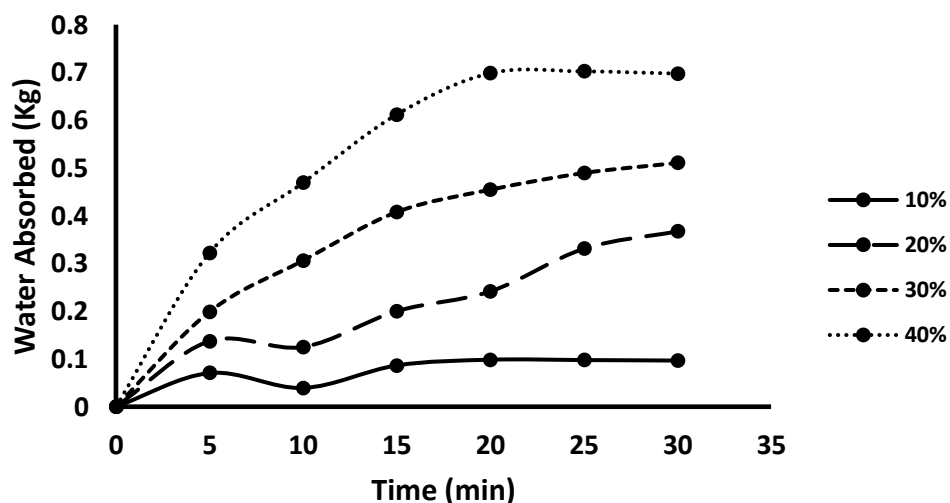


Figure 4.5-2: Capillary water absorption per unit area of lightweight geopolymer material.

Figure 4.5-2 shows that from time 0 the geopolymer absorbs water gradually but after 25 minutes of immersion in water the water uptake becomes slow, approaching a constant water absorption uptake. For 10% of waste paper content, the water absorbed within 30 minutes is the lowest among the four waste paper compositions. 10% waste paper geopolymer has the least amount of waste paper which makes the geopolymerisation process and the polycondensation stage effective, allowing the geopolymer network to form with minimal voids and air bubbles when moulding the paste. 30% and 40% waste paper absorbed the most

amount of water as waste paper creates larger voids than with the lowest amount of paper; waste paper disrupts the geopolymerisation process to build the three dimensional network effectively. 20% waste paper content, although it absorbed a higher amount than 10%, was below 30% and 40%.

Research has shown that waste paper pulp with increasing immersion time absorbs water at a rapid rate. Salem and Al-Salami (2016) reported that waste paper's cellulosic characteristics allow the waste paper pulp in papercrete to absorb a substantial amount of liquid immediately when it comes into contact with water to which, after a while, water molecules in the material are additionally conducted to fill the void spaces. It should be noted that the geopolymer only contains waste paper fibre (without any aggregates) which is a highly hygroscopic material, hence its reaction to water is obviously supposed to vary from the reaction of other materials (such as a block of masonry or concrete block) to water. As shown in Figure 4.4-2, after 15 minutes of exposure, the geopolymer absorbed a significant amount of water. This result generally indicates that the materials used in this study absorb water at a high rate by lightweight geopolymeric content.

Figure 4.5-3 shows the rate at which each waste paper content absorbs water.

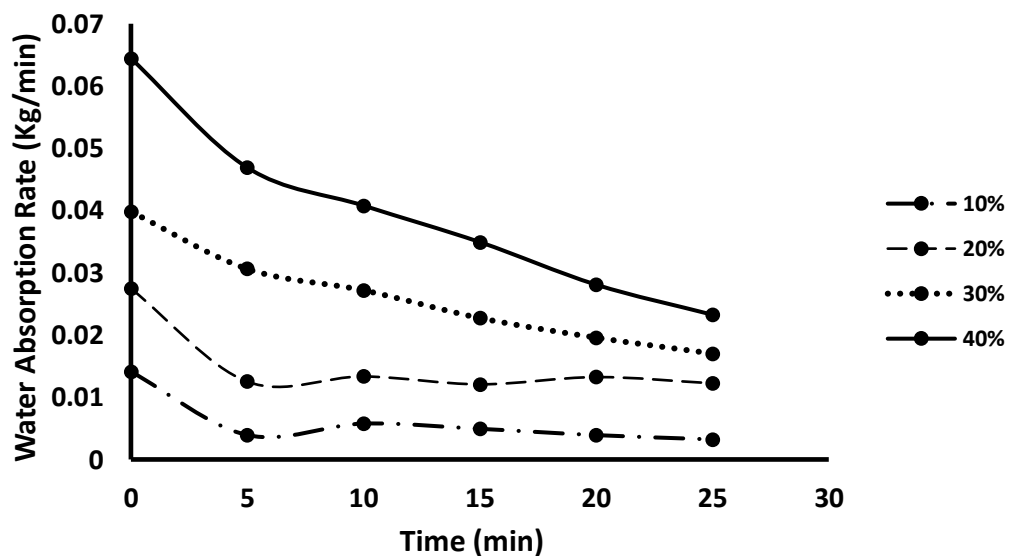


Figure 4.5-3: Capillary water absorption coefficient of lightweight geopolymer material.

For all waste paper compositions between 0 minutes and 5 minutes, the geopolymer absorbs water at a faster rate, and as the time increases the rate of absorption decreases. As mentioned above this is because the moment the geopolymer is in contact with water, it fills the voids and

cracks more quickly, and because waste paper is the filler material its properties promote the water absorption rate. A similar observation has been reported for most waste paper based blocks (papercrete) Akinwumi et al. (2014) and Yun et al. (2007) observed the same results with a papercrete block.

Therefore, due to its high capillary water absorption coefficient, it is recommended that lightweight geopolymeric content should not be used in near-ground walls (i.e. substructure). Compared to the papercrete recommendation stated to have high water absorption in previously published literature (Akinwumi et al., 2014), LWGM is recommended for use in non-load-bearing walls at least 1 m above ground level and should be mounted on a damp-proof membrane as well as covered with plastering mortar to avoid the capillary rise of water from the ground into the microstructure. The water absorption test performed on South African Lethabo graded CFA geopolymer specimens was therefore equivalent to the information available in the literature and was therefore suitable for use in the manufacture of construction materials.

The formulations developed in this work resulted in the synthesis of geopolymer materials with high compressive strengths which can be used in the manufacturing of non-loadbearing construction materials (wall partitions).

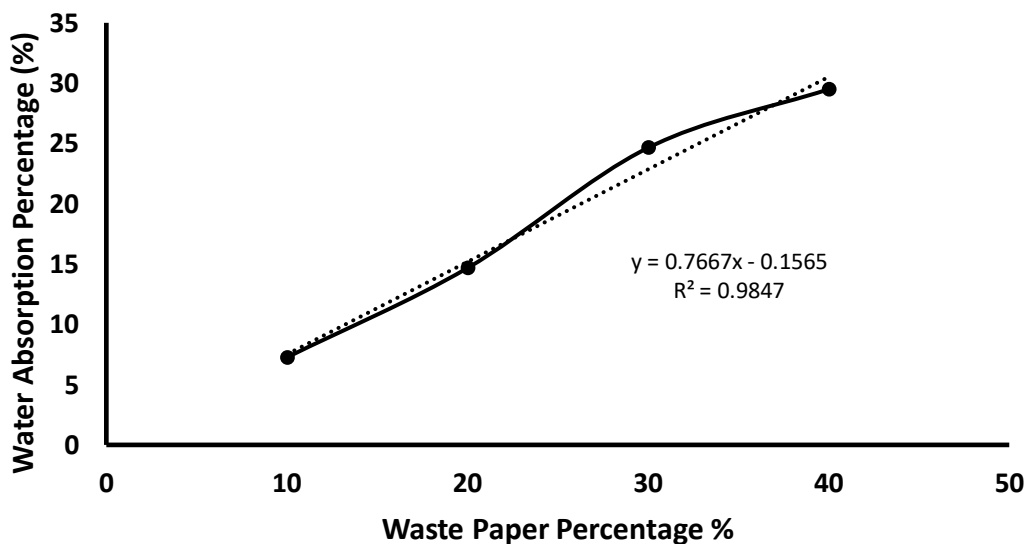


Figure 4.5-4: The effect of waste paper content on water absorption percentage.

Hence, the water absorption test was performed on the geopolymer samples in order to understand to what extent these materials would absorb water following the procedure described in chapter three (section 3.4.4).

4.6. Products Developed using Optimum Formulations and Conditions

The formulations and conditions optimised in the present study were aimed to be used in the construction industry for non-load bearing applications. The outcomes from this research have shown that CFA and waste cardboard without adding aggregates can develop a compressive strength up to 4.51 MPa at 20% waste paper content. Further, the investigations regarding the formulations, conditions, and characterisation of fly ash based geopolymer materials described in chapter four demonstrated that good quality building materials can be made out of the geopolymer formulations and conditions.

There are many building materials available in the market, including dry wall/wall partitions and papercrete bricks. These materials are traditionally made from gypsum and wood composites as well as cement, sand and paper for papercrete as shown in Chapter 2 (Section 2.10). In this study, lightweight geopolymeric bricks were made from geopolymer using the conditions and formulations developed and these bricks are ideal for indoor purposes. The same formulation was applied during the production of the wall partitions and papercrete bricks.

4.6.1. Light Weight Geopolymeric Bricks

The lightweight geopolymeric bricks were made by complying with the SANS 10164-2:2008 for masonry blocks. In this study, the production of the lightweight geopolymer bricks was carried out by mixing 2.4 kg of FA with 0.64 kg of Na_2SiO_3 , 0.32 kg of NaOH solution (10M) and 0.6 kg of waste paper for 45 minutes and shutter oil was used as a releasing agent. Then, the paste was cast in the brick mould, wrapped in a plastic sheet and put in an oven at 80°C for 24 hours. Thereafter, the paving bricks were removed from the mould and put back in the oven for 36 hours. The dimensions of the paving bricks were 220 mm in length, 110 mm in width and 49 mm in depth. The sample of the lightweight geopolymeric brick produced in this study is presented in Figure 5.2-1.



Figure 4.6-1: Light weight geopolymeric bricks produced from South African Lethabo classified coal fly ash.

Figure 5.2-1 showed the paving bricks produced in this study. Three samples of paving bricks were weighed and the average mass was 1.13 ± 0.5 kg. During the optimisation study, the lightweight geopolymer bricks developed a compressive strength of 3.25 MPa at the age of 7 days of the aging period respectively; the water absorption was 25.67%. The lightweight geopolymer bricks made in this study met certain non-load bearing building material requirements, according to SANS 1058:2012.

Therefore, good **lightweight geopolymeric** bricks were made using the geopolymer formulations and conditions developed in the present study. The durability and mechanical properties of fly ash based lightweight bricks can compete with clay and papercrete bricks available in the market. However, certain tests still need to be done on the lightweight bricks in order to comply with all relevant standards, among which is thermal conductivity. Apart from lightweight geopolymeric bricks, fly ash based wall partitions board were also produced in the present study.

4.6.2. Light Weight Geopolymer Partition Board

The CFA-based partition board was made according to SANS 542-2012 for partitioning walls. The same formulations and conditions were applied when making the lightweight geopolymer bricks (section 5.2.1). The dimensions of the sample partitioning board were 300 mm in length,

210 mm in width and 15 mm in depth. The sample board produced in this study is shown in Figure 5.2-2. Figure 5.2-2 showed the partitioning board made from coal fly ash and waste paper with an overall size of 300 x 210 mm. The mass of the board manufactured at Gyproc Saint-Gobain South Africa varied from 30 to 58 kg per square meter (Gyproc, n.d.). Thus, the use of 20% waste paper in the development of the lightweight geopolymer considerably reduced the mass of the board, implying a considerable saving in the supporting wood structure.

The fly ash based lightweight geopolymeric material and lightweight brick were made using the conditions and formulations optimised in the present study that developed sufficient mechanical properties and durability to meet specifications. The two products were made at laboratory scale and no pigments or other additives were added to these products which would be needed in order for them to be used as partitioning boards/dry wall or non-load bearing waste paper and fly ash-based brick. As mentioned above, good mechanical properties and durability for specific formulations were obtained (see Chapter 4). Therefore, it was important to investigate the cost involved during the geopolymerisation process. Thus, the cost of production is calculated in the next section.

4.7. Chapter Summary

In section 4.4 various formulations and conditions for the synthesis of lightweight geopolymeric material were investigated.

In section 4.4.1 the effect of sodium silicate on geopolymerisation process was investigated. Six levels (0, 0.5, 1, 1.5, 2, 2.5) of sodium silicate to sodium hydroxide ratio were utilised in this investigation. In Figure 4.3-1, the optimum ratio selected was 2 at which the highest compressive strength was obtained.

In section 4.4.2 the effect of NaOH concentration was investigated and 14 M had the highest compressive strength; however, the aim of this project was to use fewer chemicals to minimise cost and working to protect the earth from chemicals that would affect the environment further if they were used in excess. Although 14 M had the highest compressive strength, 12 M compressive strength is less than 14 M compressive strength by less than 5 %. 12 M can be used as the optimum for this scenario.

In section 4.3.3 the effect of curing temperature on geopolymerisation was investigated and it was observed that the compressive strength increased with an increase in temperature. The optimum temperature was 80°C because that is where the geopolymer obtained the highest compressive strength.

In section 4.3.4 the effect of compacting force was studied as the filler material was waste cardboard, and paper is known for its fibrous and high-water absorptivity qualities. The addition of pulp to the geopolymer paste resulted in the paste becoming viscous and this resulted in difficult moulding. Moulding using hands resulted in voids which affected the strength of the geopolymer. The effect of compacting force was investigated for all four waste paper contents involved in the study and it was observed that the compressive strength increased with an increase in hydraulic force and the optimum was 5 MPa.

In section 4.4.5 the effect of waste paper content on compressive strength was investigated and it was observed that the strength of the geopolymer decreased with an increase in waste paper. The results of this study were compared to construction standards and 20 % waste paper content passed the minimum requirement standards for non-load bearing construction materials. At 10 M NaOH concentration and 20% waste paper content the geopolymer passed the ASTM and SANS standards which conclude 10M to be the optimum concentration as this will save chemical usage whilst achieving the product strength.

Section 4.4.7 investigated the effect of weight on compressive strength and it was observed that the weight decreases with an increase in waste paper content. 40% waste paper content was shown to have the lowest weight but its compressive strength, as depicted in section 4.4.5, does not pass the minimum standard requirements for lightweight construction materials. Therefore it is concluded that the optimum waste paper composition was 20% as it is 50% lighter than conventional geopolymer brick.

Various conditions were investigated to determine the optimum formulation and development conditions. In section 4.4.3 the temperature used was 80°C. A further investigation was conducted at a temperature of 60°C, and the results are depicted in Figures 4.7-1 to 4.7-2.

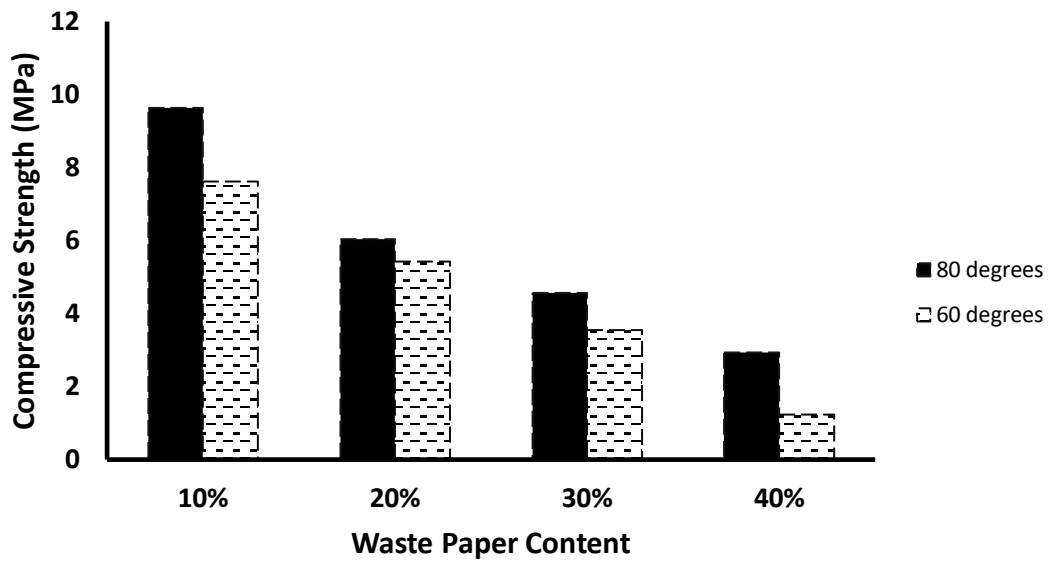


Figure 4.7-1: Compressive strength of lightweight geopolymeric material at 60°C and 80°C at 14 M NaOH concentration.

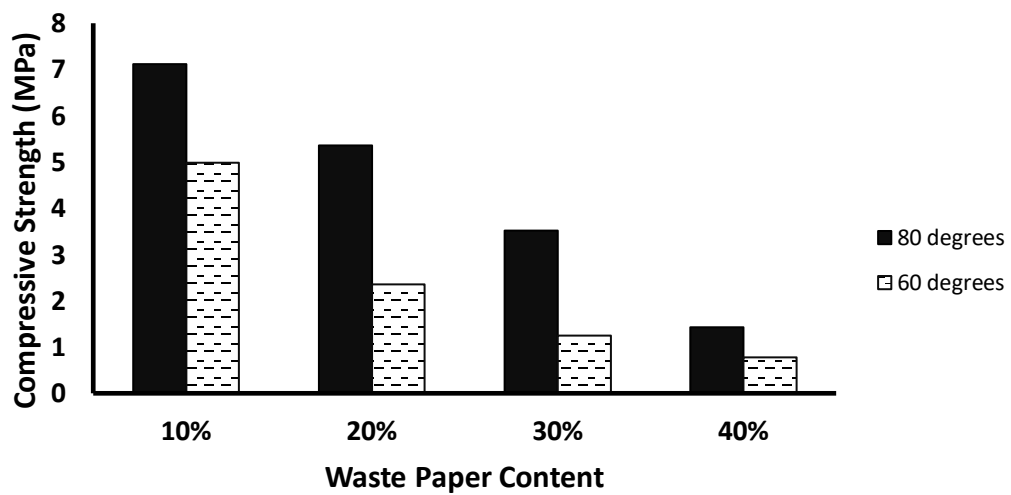


Figure 4.7-2: Compressive strength of lightweight geopolymeric material at 60°C and 80°C at 12 M NaOH concentration.

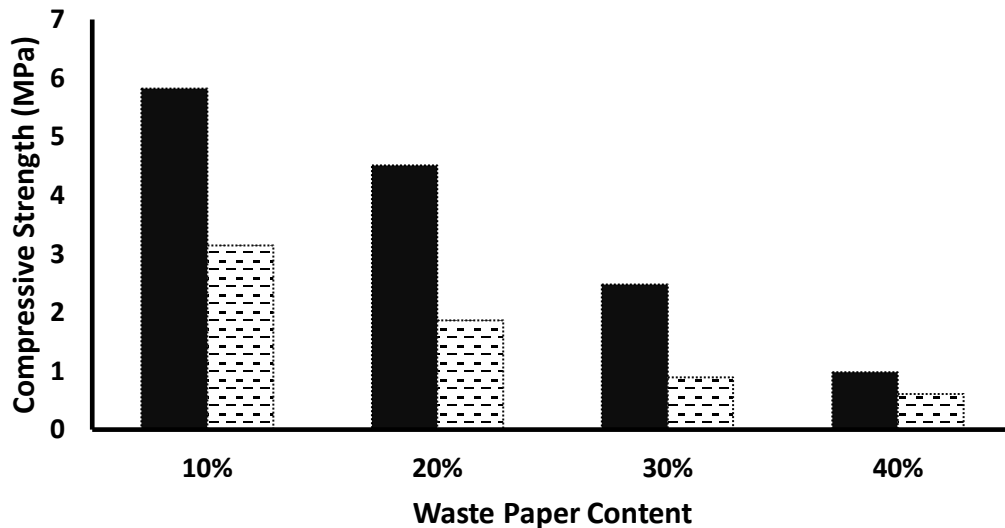


Figure 4.7-3: Compressive strength of lightweight geopolymeric material at 60°C and 80°C at 10 M NaOH concentration

In figure 4.7-1 the compressive strength test of lightweight geopolymeric material was tested at 14 M NaOH concentration. The compressive strength test ranged from 9.63 MPa 10% waste paper content to 2.94 MPa 40% waste paper content. The compressive strength decreased with an increase in waste paper content, as discussed in section 4.3.5. At 60°C the geopolymer compressive strength ranged from 7.62 MPa 10% waste paper content to 1.24 MPa at 40% waste paper content.

In figure 4.7-2 the compressive strength test of lightweight geopolymeric material was tested at 12 M NaOH concentration. The compressive strength test ranged from 7.12 MPa 10% waste paper content to 1.43 MPa 40% waste paper content. The compressive strength decreased with an increase in waste paper content as discussed in section 4.3.5. At 60°C the geopolymer compressive strength ranged from 4.98 MPa 10% waste paper content to 0.78 MPa at 40% waste paper content.

In figure 4.7-3 the compressive strength test of lightweight geopolymeric material was tested at 10 M NaOH concentration. The compressive strength test ranged from 5.82 MPa 10% waste paper content to 0.98 MPa 40% waste paper content. The compressive strength decreased with an increase in waste paper content as discussed in section 4.3.5. At 60°C the geopolymer compressive strength ranged from 3.14 MPa 10% waste paper content to 0.60 MPa at 40% waste paper content.

The lightweight geopolymer cured at 60°C was cured for 120 hours as the geopolymer had cardboard pulp with a water/paper ratio of 3.5. Despite the curing taking longer the geopolymer strength can be lower than the geopolymers cured at 60°C. Therefore, 80°C does result in a shorter curing period and attains higher strength. Thus, the results shown from 4.6.1 to 4.6.3 indicate that the compressive strengths increased with the increase in curing temperature, with decrease in waste paper content, and increase in NaOH concentration.

The formulations developed in this work can be selected and applied depending on the requirements of the manufacturing of building materials in the construction industries. The objectives of developing new formulations to make geopolymers of various compressive strengths were motivated by the idea of replacing gypsum board as well as some wood-based dry wall with industrial wastes such as coal fly ash and waste paper because they produce less CO₂ emissions and are more environmentally friendly and cost-effective. The results obtained in this work can be used to make building materials.

Chapter 5: Non-Load Bearing Construction Products and Production Cost Evaluation

5.1. Overview

The formulations and conditions developed in this study were tested using civil engineering tests assigned to non-load bearing construction materials such as a dry wall, masonry blocks, and papercrete in order to know the mechanical properties. Therefore, this chapter presents the products developed and the cost of manufacturing during the geopolymerisation process using Lethabo (South African power station) classified CFA and waste cardboard. The preliminary geopolymer products developed in this study are shown in the next section.

5.2. Material and Energy Balance

5.2.1. Material Balance in the Geopolymerisation

Different parameters involved in the synthesis of LWGM were studied and optimum conditions for each property were determined. It was concluded that the LWGM can be used as a non-load bearing construction material. However, this section is to determine the material and energy balance of the system to determine the efficiency and effectiveness of the process. The material balance is a prerequisite for accounting for input raw materials and final products. In other words, the material balance is the application of the law on the conservation of the mass, which states that the mass cannot be created or destroyed. Figure 4.6.1 shows the boundary of the total material balance. All the equations are rendered at a steady state, implying that what comes in is equivalent to what goes out. The optimum concentration of NaOH was found to be 10 M in section 4.6.2.

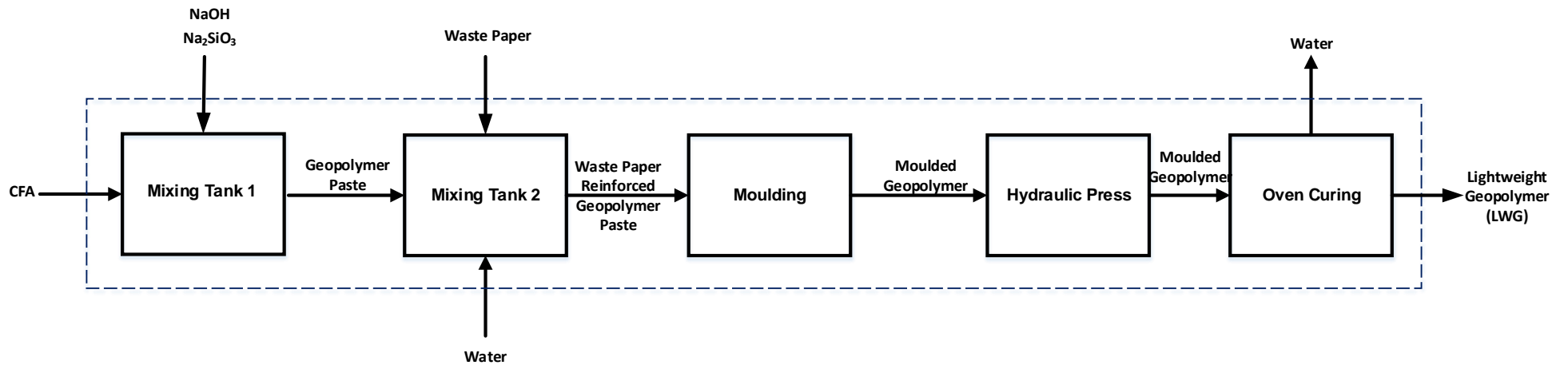


Figure 5.2-1: The process flow diagram of the geopolymer process.

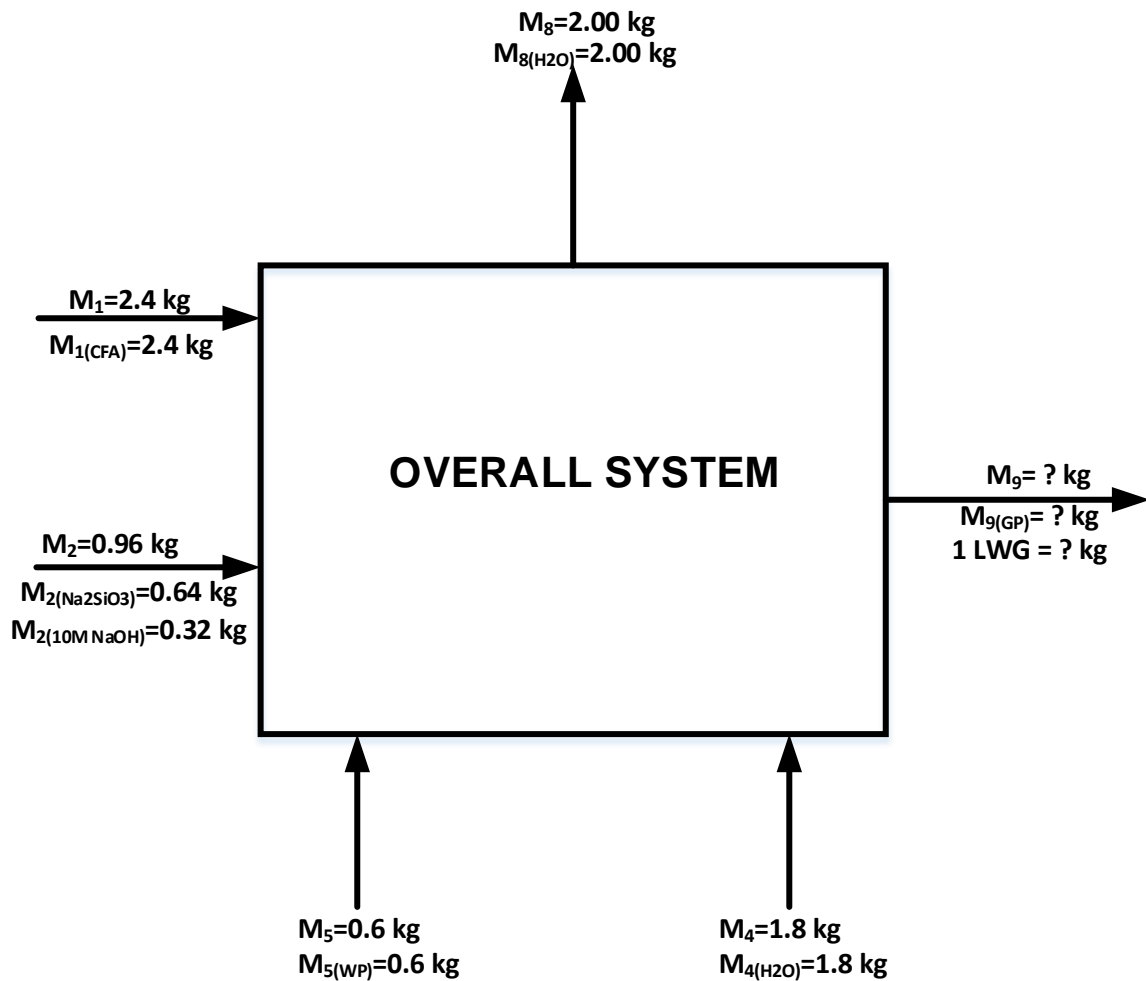


Figure 5.2-2: Overall mass balance.

Where:

M_1 = the mass of solid constituted of CFA fed to the process (kg)

M_2 = the sum of the mass alkali liquid (NaOH and Na_2SiO_3) fed to the process (kg)

M_4 = the sum of H_2O fed to the process (kg)

M_5 = the waste cardboard fed to the process (kg)

M_8 = the H_2O that has evaporated during the curing process (kg)

M_9 = Geopolymer= the total mass (in the wet form) of geopolymer produced from the process (kg)

The general material balance equation:

Accumulation = input– output + generation - consumption

Assumptions:

The system is at steady state, accumulation = 0

Input + generation = Output + Consumption

The balanced quantity is total, generation = 0 and consumption = 0

Input = Output

The theoretical total mass of 20% waste paper content geopolymer was calculated as follow:

$$M_1 + M_2 + M_4 + M_5 = M_8 + M_9$$

$$M_1 = m_{CFA} = 2.4kg$$

$$M_2 = m_{NaOHsolution} + m_{Na_2SiO_3}$$

$$0.32 + 0.64 = 0.96kg$$

$$M_4 = m_{H_2O} = 1.8kg$$

$$M_5 = m_{WP} = 0.6kg$$

% composition of NaOH and Water in NaOH solution

10M NaOH = 1kg H₂O and 0.4 kg NaOH

$$\% \text{ composition for NaOH} = \frac{0.4kg}{1.4Kg} \times 100 = 28\%$$

$$\% \text{ composition for H}_2\text{O} = \frac{1kg}{1.4Kg} \times 100 = 72\%$$

$$\therefore 0.28 \times 0.32 = 0.0896$$

$$0.72 \times 0.32 = 0.2304$$

$$M_1 + M_2 + M_4 + M_5 = 5.67$$

4% loss of moisture during manufacturing

$$0.04 \times 5.67 = 0.2268 \text{ kg before drying}$$

$$5.4 - 0.2268 = 5.1732$$

$$5.4 - 3.4 = 2 \text{ kg}$$

H_2O evaporated is 2 kg

The paste generated after the mixing stage was cast in 4 moulds of 100x100x130 (mm³) before hydraulic compression written in terms of sample and the masses are shown in Table 4.5-1.

Table 5.2-1: Mass of the cast, empty moulds and the mass of the paste

Material	Specimen 1 (kg)	Specimen 2 (kg)	Specimen 3 (kg)	Specimen 4 (kg)
Mould + paste	2.70	2.71	2.70	2.70
Mould	1.35	1.36	1.36	1.35
Actual Mass (kg)	1.35	1.35	1.34	1.35

Finally, the actual total mass was determined:

$$M_4 = \text{sample 1} + \text{sample 2} + \text{sample 3} + \text{sample 4} = 1.35 + 1.35 + 1.34 + 1.35 = 5.40 \text{ kg}$$

Figure 4.6-2 shows the overall material balance of the LWGM synthesis. Table 4.6-1 shows the actual mass of the LWGM specimen before curing. The actual total mass of a geopolymer of 5.40 kg was the mass of the paste (wet form) corresponding to M_4 . 5.67, and 5.40 kg is the theoretical and actual mass respectively of the LWGM samples respectively. The mass lost during the synthesis is the reason why the actual mass is about 95% of the theoretical mass; lost weight percent was calculated as follows:

$$\begin{aligned} \text{Total Mass lost \%} &= \frac{M_{\text{theoretical}} - M_{\text{actual}}}{M_{\text{theoretical}}} \times 100 \\ &= \frac{5.67 - 5.40}{5.67} \times 100 \\ &= 4.76 \% \end{aligned}$$

As mentioned above, the material balance was carried out in two stages, namely the mixing, and moulding. 4.76 percent weight was lost during these processes, where the starting materials switched states from slurry to paste, which implies that the weight loss could be due to the evaporation of the liquid and also to any paste that stayed attached to the mixing system wall and to the evaporation that happened during the mixing process, even though the mixer setup was sealed. Therefore, the material balance has shown that the production of geopolymer concrete has recovered 95,23 per cent of the starting material. As a result, it could be said that this process has been carried out successfully and no waste has been generated. The rule on the preservation of the mass was confirmed because the volume of the material was estimated to be 98,24%, which is almost equal to the amount of the feed.

Furthermore, as 5.40 kg of the geopolymer paste was dried in an oven at 80°C for 60 hours and some of the water had evaporated during the drying process, it was determined using the concept of moisture content in the next section.

Moisture Content Calculations

In this section, the moisture content study was carried out on the geopolymer samples produced using 10 M NaOH concentration and 20% waste paper percentages evaluated in this study. The moisture content was used to determine the water amount that evaporated from the geopolymer paste after drying the geopolymer samples in a hot air oven at 80°C for 60 hours.

The synthesis of geopolymer involved the mixing, moulding, and drying, and the final products were dried and hardened geopolymer samples. The mass of wet and dry samples is depicted in Table 4.5-1.

The equation below was used to determine the moisture content % for 20% waste paper content and the experiments were done in triplicate.

$$\%W = \left(\frac{A - B}{A} \right) \times 100$$

Where:

% W = percentage of moisture

A = Weight of wet sample

B = Weight of dry sample

	Sample 1 (kg)	Sample 2 (kg)	Sample 3 (kg)	Sample 4 (kg)
Mass_{wet}	1.35	1.36	1.36	1.35
Mass_{dry}	0.85	0.85	0.86	0.86
%W	37.03%	37.50%	36.76%	36.30%

During curing the moulds are covered with plastic to prevent moulds from losing moisture. During the study of the effect of the amount of water in this work, it was proven that water participated in the improvement of the mechanical properties of the product in the geopolymerisation process; however, for this experiment, there was a lot of water involved due to the use of waste paper as a filler material, therefore, a considerable amount of water loss is acceptable an excess of water reduced the strength of the geopolymer. The hardening of the paste was done in an oven at 80°C for 60 hours. Approximately 37 % of the water of each sample evaporated during the hydrothermal treatment in the oven. Thus, the amount of evaporated water and dried geopolymer was calculated as follows:

Total Weight before drying is 5.40 kg

Total Weight after drying is 3.40 kg

$$Water_{loss} = 5.40 \times 0.37 = 1.99 \text{ kg}$$

The amount of water evaporated is 1.99 kg. These findings conclude that from the total water content (alkali liquid and pulp water) 1.99 kg was lost through evaporation; the remainder was retained in the geopolymer and participated positively in the improvement of the mechanical properties of the inorganic polymer materials manufactured in this study. Similar observations were made by Abdullah et al. (2011), who suggested that the intensity is reduced when the removal of moisture from the specimens is permitted, and therefore it is advised that the geopolymer reaction involves the presence of moisture in order to develop good strength. Based on the results obtained in section 4.3, the loss of moisture leads to the low compressive strength properties of the lightweight geopolymer.

5.2.2. Energy Balance in the Geopolymerisation

The formulations and conditions produced in this work were made possible by the use of devices such as the mixer and the oven. The devices used electrical power. The quantification of the energy used was, therefore, a requirement for the successful operation of the geopolymerisation method. The power used by the mixing system and the oven was measured using the actual current and voltage measurements during the test.

Energy balance was done around the mixer and the drying oven as depicted in Figure 4.5-3.

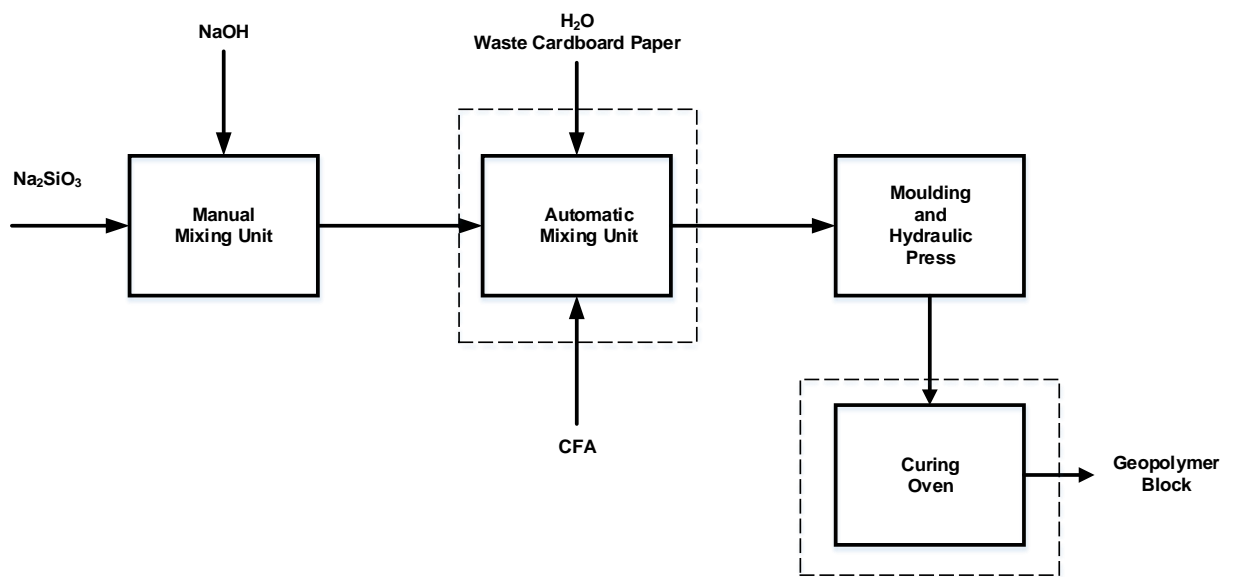


Figure 5.2-3: Energy Balance around the mixer and oven for LWGM production.

Energy consumed by the mixing unit

As shown in Chapter 3, an anchor agitator with a motor was the mixer used for mixing all the materials. The energy it consumed was determined during the mixing 20% waste paper which is 0.6 kg and 2.4 kg of FA, 0.32 kg NaOH, 0.64 kg Na_2SiO_3 and 1.8 kg of water mixed for a total time of 45 minutes.

Table 5.2-2: Mixer Description Adapted from IMER Group (2016)

Variable	Unit	Value
Machine Weight (packing)	kg	32
(w*L*h)	mm	585/630/795
Drum Diameter	mm	580
Mixer motor (230/50Hz)	kW	0.55
Mixing Speed	rpm	55
Current	A	5
Drum Size	L	56
Mixer Performance	L	47

The power required was calculated using

$$P=VI \text{ ...Equation 4.2}$$

Where,

P= power required (W)

V= voltage (V)

I= current (A)

$$\begin{aligned} P &= V \times I \\ &= 230 \times 1.5 \\ &= 345 \text{ W} \end{aligned}$$

The motor efficiency was assumed to be 75%.

$$P_e = P \times n$$

Where,

P_e = electrical power (kW)

n = efficiency (%)

P = power required (kW)

$$P = \frac{P_e}{n}$$

$$P = \frac{345}{0.75} = 460W = 0.46kW$$

Therefore, the power required was used to determine the electrical consumption.

The mixing system was used for a total time of 45 minutes, which is equal to 2700 seconds (s); the energy used by the engine was measured as follows:

$$Q = P_e \times t$$

Where,

Q= energy (kJ)

t= time (s)

$$Q = P_e \times t$$

$$= 0.46 \times 2700$$

$$= 1242 \text{ kJ}$$

The engine energy used was estimated to be 1242 kJ for 5600 g of starting materials and 45 minutes after mixing. The speed of the mixer (55 rpm) was slow and inadequate to make more than 6 kg of geopolymer paste. In order to form the geopolymer paste for all waste paper content, four separate batches had to be prepared, therefore a total mass of 22 kg of paste was made in a total time of 3 hours which is 10800 seconds. The energy required to mix 22 kg in 10800 seconds is determined below.

$$Q = P_e \times t$$

$$= 0.46 \times 10800$$

$$= 4968 \text{ kJ}$$

The estimated energy required to mix 22 kg of geopolymer paste was 4968 kJ which was equivalent to 0.9 kWh per 3 hours of use in a day.

Energy consumed by the oven during curing

The electricity used by the oven during curing was calculated using an experimental voltage and a current of 225.5 V and 4.2 A respectively. These readings are reported during the oven drying of fly ash based on geopolymer paste. The oven was big enough to fit 12 curing moulds of 100 mm³. Each waste paper content had 3 geopolymer paste and four waste paper percentages which were investigated in this study. This makes a total of 12 moulds which were calculated to be a weight of 22 kg of paste. The geopolymers were dried at 80 degrees Celsius for 60 hours. The energy used by the oven was therefore calculated as follows:

$$P=VI=225.3 \times 4.3=968.79 \text{ W}=0.96879 \text{ kW}$$

$$P=V \times I$$

$$= 225.2 \times 4.2$$

$$= 0.9460 \text{ kW}$$

$$60 \text{ hours curing} = 216 \text{ 000s}$$

Energy used for curing

$$Q= 216000 \times 0.94560 = 204 \text{ 249 kJ}$$

The oven consumed a lot of energy compared to the mixing system, a result is attributed to the disparity in current used by these two equipment systems. The overall amount of energy used in the geopolymerisation phase was determined to be the maximum of the energy consumed in the oven and in the mixing system. This was determined as follows:

$$Q_{\text{Total}} = Q_{\text{Oven}} + Q_{\text{Mixer}} = 204 \text{ 249} + 4 \text{ 968} = 209 \text{ 217 kJ}$$

The geopolymerisation process used total energy of 209 217 kJ to make 12 cubes of lightweight geopolymerisation materials.

5.3. The production cost of lightweight geopolymeric materials

The production cost of lightweight geopolymeric bricks and partitioning walls was studied in this section using the formulation of a fly ash based geopolymer activated with 10 M of NaOH because this concentration was found to be the optimum for the synthesis of geopolymer (see Chapter 4).

In this section, the cost involved in the production of lightweight geopolymeric bricks and partitioning/dry walls activated with 10 M of NaOH cured at 80°C for 60 hours was investigated. The calculations included the cost of raw materials, energy consumption of a mixer and an oven, operating labour and the cost of wall panels per square meter and lightweight geopolymeric brick per kilogram.

The material balance (section 4.4.1) demonstrated that the mass of the hardened geopolymer (3 samples) was 22 kg per day (12 samples) which was the rate of production of geopolymer in this study. For this specific study, it is important to note that waste paper and fly ash were provided for free by Gayatri Paper Company and South African Ash Resources (SAAR) respectively. Although these materials were provided free of charge, it is necessary to include the cost of transportation from these companies to the laboratories. Apostolakou et al. (2009) reported on formulas and procedures which were used in this report for calculating the cost of production of a geopolymer. The cost of production was calculated using 22 kg of geopolymeric materials per day and 5 000 kg per year, assuming the plant capacity to be laboratory scale.

5.3.1. Production Cost using a Plant Capacity of 5 000 kg/year

The cost estimate begins with the assessment of the fixed capital expenditure at a plant volume of 5000 kg/year. Fixed capital spending is a crucial variable in value production. Table 5.3-1 shows the estimate of the cost of the installed equipment (SIEC), the overall cost of the installed equipment (TIEC) and the fixed capital investment (FCI) including the value of the main equipment used for the geopolymerisation process.

Table 5.3-1: Equipment cost for the production of lightweight geopolymeric materials

Equipment	Size	Price (ZAR)
Plastic and Nylon Moulds	100 mm ³ x 15	11 500,00
Hot Air Oven (EcoTherm)	400 litres	23 396.00
Imer Mixer	56 litres	16 000.00
Sum of installed equipment		50 896
Total installed equipment cost		61 075.20
Fixed capital investment		122 150.40

The calculations were carried out as follows:

$$\text{TIEC} = 1.2 \times \text{SIEC} = 1.2 \times 50\,896 = \text{R } 61\,075.20$$

$$\text{FCI} = 2 \times \text{TIEC} = 2 \times 61\,075.20 = 122\,150.40$$

The cost of raw materials was estimated as follows:

Table 5.3-2: Raw Materials cost for the 5000kg/yr production

Raw Materials	Quantity(kg/year)	Cost (ZAR)
Sodium Hydroxide	300	700
Sodium Silicate	1200	3500
Shutter Oil	8	215
Water	3000	100
Total		4515

A few raw materials were utilised for the production of lightweight geopolymeric materials. These are sodium silicate and sodium hydroxide which were mainly for the geopolymerisation process, followed by shutter oil which is a releasing agent for the moulds. The cost of these materials was estimated in table 5.3-2 above using 1000 kg of sodium hydroxide and sodium silicate.

Cost of Utilities is as follows:

Table 5.3-3: Cost of Operational Utilities per year

Equipment	kWh/Batch	kWh/year	ZAR/kWh	Cost (ZAR/year)
Mixer	1.35	324	0.89	288.36
Oven	58.1	13 944	0.89	12 410.16
			Total	12 880.08

In this analysis, the cost of utilities was the cost of electricity. It was measured during the energy balance (section 4.4.5) of the geopolymerization cycle used by the mixer and the oven. The mixer used 0.45 kw, was used for 3 hours, and the level of energy was 1.35 kWh per day. While the oven had used 0.969 kW, it had been used for 60 hours and had an energy level of 23.25 kWh per day. The rate of electricity in South Africa is 0.89 ZAR per kWh (Source: <http://www.capetown.gov.za/Family%20and%20home/residential-utility-services/residential-electricity-services/the-cost-of-residential-electricity>, Accessed on the 30th of August 2019).

Operating Labour Costs are as follows:

Each operator earns R43.02 per hour based on the production operator (Source: https://www.payscale.com/research/ZA/Skill=Machine_Operation/Hourly_Rate, Accessed on the 30th August 2018). For the production of geopolymers, two operators are the maximum both working 5 hours per day which is equivalent to 1 200 hours per year.

Table 5.3-4: Operational Labour Cost

ZAR				
Cost	Hourly Rate	Daily Rate	Yearly Rate	Two Operators
Operator Cost	43.02	215.10	55 926	111 852

Below is Table 5.3-5 with a summary of the production costs.

Table 5.3-5: Summary of the geopolymeric material production costs

Cost Item	Calculations	ZAR	Percentage %
1. Raw materials	From material balance	4 515.00	1.61
2. Miscellaneous material	1% of FCI	1 221.50	0.48
3. Utilities	From material balance	12 880.08	2.04
(A). Variable costs	(1) + (2) + (3)	18 516.58	
4. Maintenance	10% of FCI	12 215.04	4.75
5. Operating labour	Manning estimates	111 852.00	40.70
6. Lab costs	20% of (5)	22 370.40	8.14
7. Supervision	20% of (5)	22 370.40	8.14
8. Plant overheads	50% of (5)	55 926.00	20.35
9. Capital charges	15% of FCI	18 322.56	7.13
10. Insurance, local taxes and royalties	4% of FCI	4 886.02	1.90
(B). Fixed costs	(4) + (5) + ... + (10)	128 953.06	
Direct production costs	(A) + (B)	147 469.58	
(C). General overheads + R&D	5% of the direct prod. costs	7 373.48	4.76
Yearly Production Cost= A+B+C	Subtotal (A)+(B)+(C)	154 843.12	100
Production Cost ZAR/kg	R 154 843.12/5 000 kg = ZAR 31.97/kg hardened geopolymer		

Note: FCI stands for fixed capital investment

Table 5.3-5 showed that ZAR 31.97 was the cost estimated for a kilogram of hardened geopolymer.

The geopolymerisation cycle was performed at laboratory scale, the production capacity of the geopolymer per day measured during the material flow was 22 kg; this production rate was expected to have a potential of 5 000 kg/year. The cost of production was high because the operation was carried out on a small scale.

The factors that made the system costly on a small scale were the manufacturing labour costs and the utility costs of the energy consumed by the mixer and the oven. Such variables do not rely very much on the rate of production and are often constant regardless of the size of the factory. Furthermore, if a bigger plant is used, the cost of purchasing the machinery would definitely increase. It would therefore be necessary to increase production efficiency in order to reduce the cost of the product.

Based on the cost of development of a geopolymer with a plant capacity of 5 000 kg/year, a scale-up study was carried out using a plant capacity of 150 000 kg/year as shown in the next paragraph.

5.3.2. Scale-up of the Geopolymerisation Process

The purpose of scaling up the geopolymerisation process would be to reduce the cost of production. Previously, 12 geopolymer samples equivalent to 22 kg (size: 100 mm³ per sample) were made and the plant capacity per year was 5 000 kg. In this section, the production cost of the geopolymer materials, if produced on a larger scale, was estimated.

The formulations and conditions used to estimate the cost of production of geopolymer at 200 000 kg/year were the same as at 5 000 kg/year. At 5 000 kg/year, the geopolymer was produced using 2.4 kg of FA, 1.8 kg of water, 0.6 kg of WP, 0.64 kg of Na₂SiO₃ and 0.32 kg of NaOH of solution (10 M). Then, it was mixed for 45 minutes. The geopolymer paste was cast in 3 moulds (size: 100 mm³) and wrapped in plastic sheet aged for 24 hours, thereafter, heated in an oven at 80°C for 60 hours.

In this section, a larger mixer (size: 820 L) and a hot air dryer oven (with 20 trays and 12 samples per tray) were used as a basis to calculate the costs for the production of a geopolymer, in this scenario, 240 samples (size: 100 mm³). The scale-up should have a plant capacity of 150 000 kg/year of geopolymer, which should be equivalent to 90 000 samples of geopolymer (size: 100 mm³) per year. The calculations of the production cost of the scale-up plant were carried out as shown below. The fixed capital investment of the scaled-up plant was calculated below. The cost of equipment and FCI are presented in Table 5.6 below.

Note: All the terms used in this section were defined in previous calculations Table 5.3-6: FCI for large scale plant capacity of 150 000kg/Yr

Equipment	Size	Price (ZAR)
Plastic and Nylon Moulds	100 mm ³ x 240 samples	180 000.00
Hot air Oven (EcoTherm)	-	75 996.00
Imer Mixer	820 litres	28 500.00
Total installed equipment cost		341 395.20
Sum of installed equipment		284 496.00
Fixed capital investment		682 790.40

The scaled-up plant would need a fixed capital investment of R 682 790.40. Thereafter, the cost of raw material for the geopolymer production was estimated as shown below.

Cost estimation of raw materials used at the scaled up plant was done for producing 150 000 kg/year of geopolymer. The number of raw materials used to produce (150 000 kg/year) of geopolymer was estimated by referring to the material balance (section 4.4.1) determined at the plant capacity of 5 000 kg/year (section 5.3.1) as follows:

Sodium silicate: 2.56 kg of Na₂SiO₃ was used to produce 22 kg/day of hardened geopolymer, thus, for a plant capacity of 150 000 kg/year of lightweight geopolymer geopolymer, the amount of Na₂SiO₃ estimated was 17 454 kg/year.

Sodium hydroxide: 1.28 kg of NaOH pellets were used to make 22 kg/day of hardened geopolymer, thus for a plant capacity of 150 000 kg/year of lightweight geopolymer, the amount of NaOH estimated was 8 727 kg/year.

Water: 7.2l of water was used to make 22 kg/day of hardened geopolymer, thus for a plant capacity of 150 000 kg/year of lightweight geopolymer, the amount of water estimated was 49 090 kg/year.

Shutter oil: 0.03 kg of the releasing agent was estimated to be used in the production of 22 kg/day, thus for a plant capacity of 150 000 kg/year of lightweight geopolymer, the amount of shutter oil estimated was 2045 kg/year.

However, CFA and waste cardboard pulp were not included in the cost estimation calculations because it was assumed to be supplied and delivered for free to the plant by South African coal power stations and waste paper recycling companies or corrugated packaging companies.

Table 5.3-7: Cost of raw materials for the production of geopolymer using a plant capacity of 150 000 kg/year

Raw Materials	Quantity(kg/year)	Cost (ZAR)
Sodium Hydroxide	8 727	18 119.57
Sodium Silicate	17 454	58 695.01
Shutter Oil	45	1 100.12
Water	90 000	3 000
	Total	80 914.70

The cost estimated of raw materials was R 80 914.70 per year for the production of 150 000 kg/year of geopolymer.

The cost of utilities

The energy used by the new mixer and oven was estimated by referring to the energy balance (section 4.4.5) investigated at the small scale (5 000 kg/year) as follows:

For the oven: the maximum power that can be used by the oven was 1 kW and the power that was consumed as calculated during the energy balance was 0.97 kW. The oven used for the scaled-up plant has a maximum power of 0.9 kW, therefore, the estimated power consumed by this oven was 0.87 kW in the production of lightweight geopolymeric materials. The oven would be used for 60 hours per batch and the rate estimated was 52.2 kWh.

For the mixer: the maximum power that can be used by the mixer was equivalent to 2.7 kWh because it was used for 3 hours per day on a small scale. The mixer used for the scaled-up plant has a maximum power of 7.5 kW, therefore, the estimated power consumed by this mixer was 6.14 kWh in the production of geopolymers. The mixer would be used for two hours per day and the rate estimated was 20.25 kWh.

Table 5.3-8: Cost of Utilities in the production of 150 000 kg/year

Equipment	kWh/Batch	kWh/year	ZAR/kWh	Cost (ZAR/year)
Mixer	20.25	4 860	0.89	4 325.40
Oven	52.20	12 048	0.89	11 045.04
			Total	15 370.44

Operating labour costs are as follows:

Each operator earns R43.02 per hour based on the production operator according to payscale 2018 (For the production of geopolymetric materials, two operators are the maximum both working 5 to 6 hours per day which is equal to 1 200 hours annually).

Table 5.3-9: Operational Labour Cost

ZAR				
Cost	Hourly Rate	Daily Rate	Yearly Rate	4 Operators
Operator Cost	43.02	215.10	55 926	223 704

Summary of the geopolymetric material production costs

Table 5.3-10 below shows that the production of 150 000 kg/year of lightweight geopolymetric materials results in 1kg of lightweight geopolymetric materials manufacturing cost of ZAR 5.06.

Table 5.3-10: Summary of the geopolymeric material production costs

Cost Item	Calculations	ZAR	Percentage %
1. Raw materials	From material balance	80 914.70	1.61
2. Miscellaneous material	1% of FCI	6 827.91	0.48
3. Utilities	From material balance	15 370.44	2.04
(A). Variable costs	(1) +(2) +(3)	103 113.05	
4.Maintenance	10% of FCI	68 279.04	4.75
5. Operating labour	Manning estimates	223 704.00	40.70
6. Lab costs	20% of (5)	44 740.80	8.14
7. Supervision	20% of (5)	44 740.80	8.14
8. Plant overheads	50% of (5)	111 852.00	20.35
9. Capital charges	15% of FCI	102 418.56	7.13
10. Insurance, local taxes and royalties	4% of FCI	27 311.62	1.90
(B). Fixed costs	(4) +(5) +...+(10)	623 046.82	
Direct production costs	(A) + (B)	726 519.87	
(C). General overheads + R&D	5% of the direct prod. costs	36 175.99	4.76
Yearly Production Cost= A+B+C	Subtotal (A)+(B)+(C)	761 335.86	100
Production Cost ZAR/kg	R 761 335.86/150 000 kg = ZAR 5.06/kg		

Thus, the main cost items for both plant capacities are presented in Table 5.11.

The difference in production cost between the lab scale and the scale-up plant was compared using a plant capacity of 5 000kg/year and 150 000 kg/year in Table 5.3-11 table below. The purpose of comparing two plant capacities for the production of geopolymer was to

understand the production cost implications and benefits of using a smaller or larger plant scale. Therefore, the manufacturing cost of inorganic polymer using plant capacities of 5 480 kg/year and 152 880 kg/year was calculated in the previous sections.

Table 5.3-11: Comparison between lab production and plant based production

Cost Item	Calculations	Lab Based	Plant Based
1. Raw materials	From material balance	4 415.00	77 914.70
2. Miscellaneous material	1% of FCI	1 221.50	6 827.91
3. Utilities	From material balance	12 880.08	15 370.44
(A). Variable costs	(1) + (2) + (3)	18 516.58	103 113.05
4. Maintenance	10% of FCI	12 215.04	68 279.04
5. Operating labour	Manning estimates	111 852.00	223 704.00
6. Lab costs	20% of (5)	22 370.40	44 740.80
7. Supervision	20% of (5)	22 370.40	44 740.80
8. Plant overheads	50% of (5)	55 926.00	111 852.00
9. Capital charges	15% of FCI	18 322.56	102 418.56
10. Insurance, local taxes and royalties	4% of FCI	4 886.02	27 311.62
(B). Fixed costs	(4) + (5) + ... + (10)	128 953.06	623 046.82
Direct production costs	(A) + (B)	147 469.58	726 519.87
(C). General overheads + R&D costs	5% of the direct prod.	7 373.48	36 175.99
Annual production cost= A+B+C Subtotal (A)+(B)+(C)		154 843.12	761 335.86
Price/kg		R31.97	R 5.06

Table 5.3-11 showed the difference in cost items using the plant capacities of 5 000 kg/year and 150 000 kg/year. A greater amount of money would be spent on large scaled up plant

compared to the smaller scale; however, it was found that the production cost using 5 000 kg/year or 150 000 kg/year plant capacity would be R 30.97/kg or R5.06/kg respectively.

Throughout the present study, it was demonstrated that the increase of the size of the mixer and oven would result in the production of a much larger volume of geopolymer but would not result in a big difference in terms of energy consumptions and would not affect the cost of the utilities much. However, the fixed capital investment would be higher at the scaled-up plant than at the laboratory scale plant due to the difference in the size of the equipment. The pay scale and working hours would remain constant but four production operators would be needed at the scaled-up plant whereas only two were necessary at laboratory scale.

In this section, it was shown that the larger the capacity of the production plant the lower the cost of the product. The cost of production could be lowered further by using a plant with a production capacity larger than 150 000 kg/year of geopolymer.

5.3.3. Cash Flow of a Plant Capacity of 150 000kg/year

Figure 5.3-1 illustrates the cumulative cash flow diagram of a plant with a capacity of 150 000kg/year.

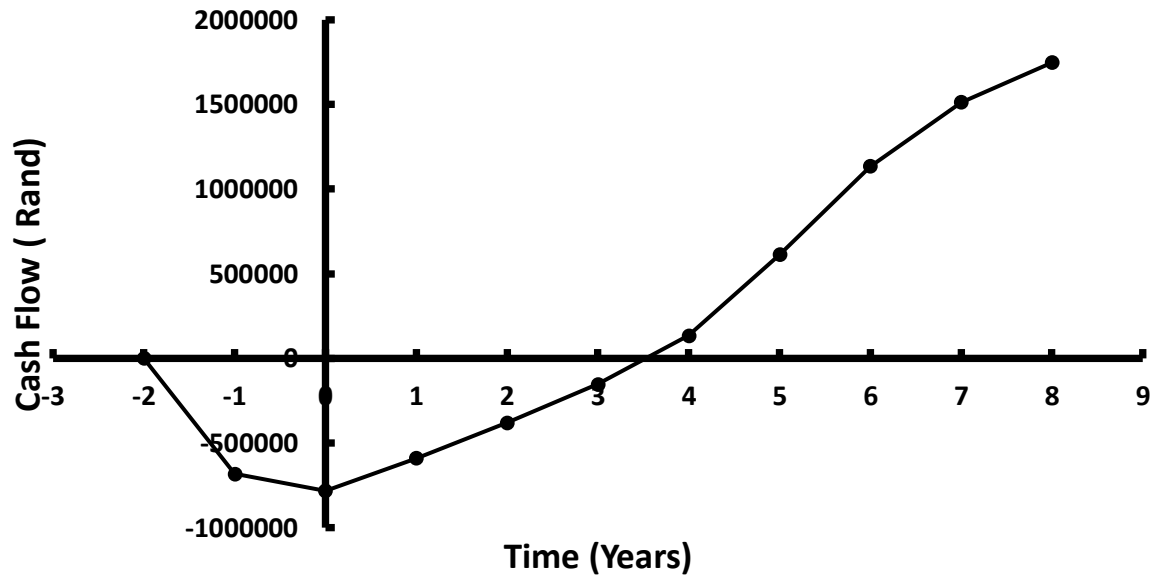


Figure 5.3-1: Cash flow of a plant with capacity 150 000kg/year

$$\text{Total NPW of project} = \sum_{n=1}^{n=t} \frac{NFW}{(1+r)^n} = \text{R } 401375.70$$

List of assumptions

- The cost associated with land is negligible
- 10% sales increase in the first couple of years is expected followed by 30% and 40% in the 3rd and 4th years respectively.
- In the last 2 years of the project there is a drop of 5% in revenue
- The process is run at steady state conditions
- The plant operates for 240 days in a year
- The salvage value of the project is negligible

Figure 5.3-1 shows the cash flow diagram for the manufacturing plant producing 150 000kg/year of geopolymeric material. ZAR 782 790.40 is the amount of money that needs to be invested in the building of the plant. Towards the end of the project, inventory and maintenance expenses are expected to continue to increase due to infrastructure wear and tear as revenues decline. The project will take 3.5 years to recover and break even (Figure 5.3-1) that was spent from the start of the project, while the first gain is shown to have been generated over the last 4.5 years of the project. The payback analysis does not consider the time value of money.

The Net Present Value (NPV) which reflects the present value of money from future cash flows is an effective way to analyze productivity. The NPV is estimated to be R 401 375 for the company's necessary rate of return of 18%. If NPV is negative, the project has to subtract value from the firm and therefore it has to be rejected as a result. On the other hand, a positive NPV implies that the plan meets the expected cost or that the project is sustainable. In order to make the venture more competitive, it is recommended that the organization should lower its necessary IRR. A lower rate of return implies earning capital at a lower rate, but the chance of making a profit is greater.

5.3.4. Commercial Dry Wall versus Experimental Dry Wall

The aim of this study is to develop lightweight geopolymeric material which will be used for non-load bearing construction materials like dry wall/wall partitioning boards. Below is table 5.3-12 which shows the difference between commercial dry wall and the experimental board.

Table 5.3-12: Commercial Dry Wall versus Experimental

Usage	Commercial Dry Wall	Fly Ash Based Dry Wall
Compressive Strength	2.5 MPa	3.05 MPa
Flexural Strength	400N to 800N	400N
Split tensile Strength	1.02 MPa	0.98 MPa
Weight	30-58 kg/m ²	40-50 kg/m ²
Density	600 kg/m ³ – 1 500 kg/m ³	685 kg/m ³
Ease of installation	Dry system – keep the site clean	Dry system
Services	Services can be easily installed	Services can be easily installed
Rates	R146.00 – R1023.99	R 200.00
Construction time	40 m ² per labour per day	40 m ² per labour per day
Modification	Can be installed in any area inside a building	Can be installed in any area inside a building

Table 5.3-12 shows that the weight per square meter, compressive strength, flexural strength, density and the price of the experimental board are all in the same range as the commercial dry wall board. The above table shows that the experimental product is, in fact, a competing product that can be used for non-load bearing construction materials for exactly the same application as the gypsum board is used for.

Chapter 6: Conclusion and Recommendation

6.1. Research summary

The aim of this study was to investigate the possibility of a lightweight geopolymeric material using waste materials such as CFA from power plants and cardboard waste paper as a filler material. The lightweight geopolymeric material was synthesised without the addition of aggregates such as sand, cement or any other alkali material. Waste paper used in this study was not incinerated and was used in pulp state.

The formulations used for this study consisted of waste paper (10%, 20%, 30% and 40% content of coal fly ash). The total weight of the dry solids was 3 kg, therefore the weight of CFA varied according to waste paper content. Sodium silicate and sodium hydroxide also varied according to the amount of CFA for each batch. The curing temperatures were 60°C and 80°C for 60 hours and the blocks were tested at the age of 7 days for compressive strength, tensile strength, flexural, water absorption and weight.

Achieving the aim of this study will aid in environmental problems caused by the materials being used for synthesis. Many problems can be resolved by the findings of this study, among which:

- The utilisation of waste paper results in the reduced amount of waste cardboard paper which is being disposed of in landfills.
- Reduced costs compared to gypsum-based dry wall.
- The reduction of CFA disposed of by South African power plants as it is used as the main source of silica and aluminium.
- No cement or sand added, resulting in less cement being manufactured.
- Replacement of cement by CFA in the construction industry.
- Replacement of gypsum boards by waste paper and CFA-based boards.
- Minimum costs required to develop the geopolymer.

These objectives were achieved by answering the research questions:

Question 1: What is the relationship between the WP:FA ratio to compressive strength?

The compressive strength decreases with an increase in waste paper content. The highest compressive strength obtained at using 10 M NaOH varied from 5.82MPa (at 10% waste paper

content) to 0.98 MPa (at 40% waste paper content), followed by 12M NaOH which varied from 7.92 MPa (at 10% waste paper content) to 1.43 MPa (at 40% waste paper content). The last one was 14 M which had 9.66 MPa (at 10% waste paper content) as the highest and the lowest 2.94 MPa (at 40% waste paper content).

These new formulations are successful because they involved the use of a large amount of coal fly ash and waste paper with fewer chemicals while developing comparative strength as commercialised construction materials. The curing temperature affects the compressive strength positively; as the curing temperature increased from 60 to 80 degrees Celsius the compressive strength increased.

Question 2: Does the water content affect the geopolymer compressive strength and shrinkage?

An increase in water content proved to improve the geopolymer strength from 4.02 MPa at 0.9 kg of water to 4.51 MPa at 1.8 kg of water; from 4.51 MPa at 1.8 kg of water to the geopolymer compressive strength decreased to 3.10 MPa at 2.70 kg of water content geopolymer.

As water content increased the shrinkage percentage increased as well for all waste paper content ratios. At 20% waste paper content, the shrinkage percentage increased from 8% at 0.9kg of water to 23% at 3.6 kg of water.

Question 3: What are the properties (mass, density, water absorption) and the economics of the synthesised lightweight geopolymeric material?

Waste paper based geopolymeric material is lighter than geopolymer concretes with a mass varying from 0.98 kg to 1.1 kg to 1.8 kg per 1000 L and density in the range of 630-850 kg/m³. The geopolymer water absorption rate ranges from 0.01 kg/min to 0.06 kg/min, which is quite a fast rate.

The estimation of the operating cost was determined using two plant capacities (5 000 kg/year and 180 000 kg/year) of the geopolymer. The results demonstrated that 1 kg of geopolymer is about R 30/kg or R10.62/kg using a plant capacity of 5 000 kg/year or 180 000 kg/year respectively. It was observed that the larger the capacity of the production plant, the lower the cost of the geopolymer product.

Question 4: Where in the construction industry can the lightweight geopolymer be used?

This lightweight construction material can be used in the construction industry for non-load bearing purposes such as wall partitioning. The formulations and conditions used in this study can be used to manufacture different products/building materials depending on the application. The optimum conditions of this study were only used to manufacture samples of dry walls at lab scale and the same formulation can be used to manufacture in house furniture such as office desks and work stations as well as cupboards.

6.2. Study Conclusion

In Chapter one section 1.2 the research problem was stated. The findings of the current study proved that CFA and WP can be used in manufacturing various building materials. A large amount of coal fly ash and waste paper was used in this study up to 70%, and merely 30% of the formulation consisted of sodium silicate and sodium hydroxide and additional water. No aggregates, sand or cement were used in any of the formulations developed in this study. The significance of this study can be summarised as follows:

A total of 84 formulations with various conditions were developed in this study, and each one of them had a specific strength and could be applied for the manufacturing of a specific product in the construction industry. The optimum formulations and conditions applied for the synthesis of lightweight geopolymeric materials consisted of 1.8 kg of water, 0.64 kg of Na_2SiO_3 , 2.4 kg of coal fly ash, 0.6 kg of waste paper, 0.32 kg of NaOH solution which were mixed for 45 minutes; when cured at 80°C for 60 hours and then aged for 7 days, it developed a compressive strength of 6.03 ± 1.04 , 5.36 ± 1.22 and 4.51 ± 1.45 for the geopolymer activated with 10, 12 and 14 M of NaOH respectively.

The wall partitioning boards and bricks samples made were developed using the above mentioned formulation. The mass of the wall was calculated to be between 30kg and 40kg per square meter and the brick was weighed to be 1.1 ± 0.5 kg, at a size of 220 mm in length, 110 mm in width and 49 mm in depth.

6.3. Findings of the Study

The finding of this study are listed below:

- The final product does not contain aggregates such as cement or sand, therefore the final product's application in the construction industry is for non-load bearing applications only.

6.4. Recommendations

The aims and objectives of this study have been achieved; however, there are several recommendations that can be made to improve and to expand the study further.

The recommendations for future studies are listed below:

- Further investigations such as thermal conductivity, acoustic properties, modulus of elasticity and flammability can be done using the same formulations and conditions developed in this current study.
- The same formulation and parameters can be investigated to find further applications of this board.
- Parameters such as hydraulic pressure, water, and curing temperatures can be optimised further depending on the applications of the board. Curing time can be reduced from 60 hours and hydraulic pressure can be reduced or increased as well. Aging days can vary from day 1 to day 90.
- The formulations in this study can be further used and tested for other products such as in house furniture, including cupboards, and in automobiles.

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Appendix A: Sample Calculation

Sample calculations for density at 20% waste paper content

$$\begin{aligned}\rho &= \frac{m}{V} \\ &= \frac{0.813}{0.00098} \\ &= 829.5 \text{ kg/m}^3\end{aligned}$$

Sample calculations for water absorbed at 20% waste paper content

$$\begin{aligned}W_{ab} &= M_f - M_i \\ &= 0.9899 - 0.8526 \\ &= 0.1373 \text{ kg}\end{aligned}$$

0.1373 kg of water absorbed after 5 minutes of water immersion.

Sample calculations for water absorption rate

$$\begin{aligned}W_{abrate} &= \frac{W_{abs}}{t} \\ &= \frac{0.1373}{5} \\ &= 0.02746 \text{ kg/min}\end{aligned}$$

Appendix B: Raw Data

0% 80°C 10 M	
Sample 1	56.92
Sample 2	50.78
Sample 3	57.67
Average	55.12
St. Deviation	3.78

10%	
Sample 1	4.84
Sample 2	4.76
Sample 3	4.85
Average	4.82
St. Deviation	0.05

20%	
Sample 1	3.15
Sample 2	3.23
Sample 3	3.08
Average	3.15
St. Deviation	0.08

30%	
Sample 1	1.98
Sample 2	2.03
Sample 3	1.89
Average	1.97
St. Deviation	0.07

40%	
Sample 1	0.98
Sample 2	1.12
Sample 3	1.23
Average	1.11
St. Deviation	0.13

0% 80 12 M	
Sample 1	79.92
Sample 2	77.57
Sample 3	78.06
Average	78.52
St. Deviation	1.24

10%	
Sample 1	6.23
Sample 2	6.07
Sample 3	6.07
Average	6.12
St. Deviation	0.09

20%	
Sample 1	4.39
Sample 2	4.33
Sample 3	4.35
Average	4.36
St. Deviation	0.03

30%	
Sample 1	2.89
Sample 2	2.97
Sample 3	2.98
Average	2.95
St. Deviation	0.05

40%	
Sample 1	1.58
Sample 2	1.96
Sample 3	1.96
Average	1.83
St. Deviation	0.22

0% 80 14 M	
Sample 1	79.92
Sample 2	77.57
Sample 3	78.06
Average	78.52
St. Deviation	1.24

10%	
Sample 1	8.23
Sample 2	8.05
Sample 3	8.15
Average	8.14
St. Deviation	0.09

20%	
Sample 1	6.07
Sample 2	6.12
Sample 3	5.98
Average	6.06
St. Deviation	0.07

30%	
Sample 1	4.87
Sample 2	4.56
Sample 3	4.87
Average	4.77
St. Deviation	0.18

40%	
Sample 1	2.56
Sample 2	2.89
Sample 3	2.85
Average	2.77
St. Deviation	0.18

Tensile Strength (MPa)					
0% 80°C 10 M		0% 80°C 12 M		0% 80°C 14 M	
Sample 1		Sample 1		Sample 1	
Sample 2		Sample 2		Sample 2	
Sample 3		Sample 3		Sample 3	
Average		Average		Average	
St. Deviation		St. Deviation		St. Deviation	
10%		10%		10%	
Sample 1	0.5428	Sample 1	0.7567	Sample 1	1.2544
Sample 2	0.5677	Sample 2	0.8045	Sample 2	1.2847
Sample 3	0.5258	Sample 3	0.7547	Sample 3	1.2454
Average	0.545433	Average	0.77	Average	1.26
St. Deviation	0.021074	St. Deviation	0.03	St. Deviation	0.02
20%		20%		20%	
Sample 1	0.6587	Sample 1	0.8127	Sample 1	1.3214
Sample 2	0.6857	Sample 2	0.8367	Sample 2	1.3104
Sample 3	0.6975	Sample 3	0.8124	Sample 3	1.2983
Average	0.680633	Average	0.82	Average	1.31
St. Deviation	0.01989	St. Deviation	0.01	St. Deviation	0.01
30%		30%		30%	
Sample 1	0.6247	Sample 1	0.7813	Sample 1	1.1894
Sample 2	0.6012	Sample 2	0.7736	Sample 2	1.1762
Sample 3	0.6289	Sample 3	0.7833	Sample 3	1.1857
Average	0.618267	Average	0.78	Average	1.18
St. Deviation	0.014929	St. Deviation	0.01	St. Deviation	0.01
40%		40%		40%	
Sample 1	0.5478	Sample 1	0.7201	Sample 1	0.9856
Sample 2	0.5542	Sample 2	0.7139	Sample 2	1.1021
Sample 3	0.5671	Sample 3	0.7116	Sample 3	0.9951
Average	0.556367	Average	0.72	Average	1.03
St. Deviation	0.009831	St. Deviation	0.00	St. Deviation	0.06

Water Absorption kg/min at 10M		Water Absorption %	
0%		0%	
Sample 1		Sample 1	4.26
Sample 2		Sample 2	3.98
Sample 3		Sample 3	4.32
Average		Average	4.186666667
St. Deviation		St. Deviation	0.181475435
10%		10%	
Sample 1	0.0033	Sample 1	7.52
Sample 2	0.0033	Sample 2	6.89
Sample 3	0.0034	Sample 3	7.25
Average	0.00333333	Average	7.22
St. Deviation	5.7735E-05	St. Deviation	0.316069613
20%		20%	
Sample 1	0.01289	Sample 1	19.11
Sample 2	0.01301	Sample 2	19.72
Sample 3	0.01223	Sample 3	18.04
Average	0.01271	Average	18.95666667
St. Deviation	0.00042	St. Deviation	0.850431263
30%		30%	
Sample 1	0.01765	Sample 1	23.47
Sample 2	0.01698	Sample 2	25.24
Sample 3	0.017254	Sample 3	24.61
Average	0.01729467	Average	24.44
St. Deviation		St. Deviation	0.897162193
40%		40%	
Sample 1	0.02345	Sample 1	29.54
Sample 2	0.02458	Sample 2	28.78
Sample 3	0.02322	Sample 3	31.45
Average	0.02375	Average	29.92333333
St. Deviation	0.00072794	St. Deviation	1.375657419

Appendix C: Publications

eaeres.org/siteadmin/upload/4566EAP1119252.pdf

17th JOHANNESBURG Infi Conference on Science, Engineering, Technology & Waste Management (SETWM-19) Nov. 18-19, 2019 Johannesburg (S.A.)

Development of Light-weight Geopolymeric Materials using Fly Ash and Waste Paper

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Abstract— The aim of this paper is to utilize coal fly ash and waste paper which are currently causing disposal problems, for the production of lightweight materials which might find application in construction industry. The view is to investigate the effects of waste-paper to fly ash ratio on the properties of lightweight, high strength geopolymeric materials produced from these wastes. The results of this study show that decreases in weight, compressive strength and tensile strength as the waste paper content increases. It can be shown that with up to 50% decrease in weight of geopolymeric material with a comparative compressive strength of 4.51 MPa, similar to commercially available construction materials can be achieved by using 20% waste paper with coal fly ash. The capillary water absorption revealed that the material absorbs water at a fast rate suggesting its suitability for indoor applications. The results of this study would provide an alternative and cost-effective approach to manufacturing of non-load bearing construction materials from waste.

Keywords— Coal Fly Ash, Lightweight, Geopolymer, Waste Paper.

applications such as an adsorbent for treatment of acid mine water, zeolites manufacturing, construction applications and recently it has been mostly used to make geopolymer[8]. The activation of aluminosilicate material by alkali silicate forming three dimensional aluminosilicate binder is called geopolymerisation and the three-dimensional binder formed is called a geopolymer [9].

To improve mechanical properties such as fire resistance characteristics, flexibility and ductability in geopolymers reinforcement of different fibres (synthetic and natural) from waste materials has been introduced [9] [10]. Waste paper has drawn interest as a reinforcement material for geopolymer due to its properties such as low density, low cost, desirable strength to weight ratio and high availability[11]. Disposal of waste paper on open dump landfills leads to environmental problems such as the pollution of soil, surface and water [12]. Waste paper has attracted the construction industry and has been employed for various purposes such as, fibre cement