

**INFLUENCE OF CASSAVA PEEL ASH AND METAKAOLIN BASED
GEOPOLYMER MORTAR ON THE BOND STRENGTH OF CONCRETE**

BY

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MTECH/SET/2018/8044

**DEPARTMENT OF BUILDING
FEDERAL UNIVERSITY OF TECHNOLOGY MINNA**

APRIL, 2023

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**A PROPOSAL SUBMITTED TO THE DEPARTMENT OF BUILDING,
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ABSTRACT

The critical element for sustainable growth in the construction industry is the development of alternative cements. A new technological process called geopolymerization provides an innovative solution, and the presence of aluminum and silicon oxides in pozzolans has encouraged its use as a source material. The present study investigated the material's properties, practices and bonding properties between geopolymeric repair material (mortar) and concrete substrate. An experimental program was executed to establish a connection between the alkaline activator composition and the properties of geopolymer mortar and concrete in fresh and hardened states. Concentrations of sodium hydroxide and sodium silicate were ascertained that are advantageous for constructability and physical properties. Also, the influence of the properties of the repair materials itself and also the surface preparations of concrete substrate on bond strength was evaluated. The factors affecting the bond strength was studied through slant shear tests made on 72 specimens. The CPA/MK based mortar is noticeably more flow-able/workable in comparison with equivalent cement-based mortar. The initial and final setting time in 50%CPA/50%MK, 100%CPA/0%MK geopolymer repair mortar is considerably shorter than cement-based mortar, which can be in most cases beneficial for using this type of mortar in repairing concrete structures. 100%CPA/0%MK geopolymer mix delays the initial setting time to 25 minutes, and the final setting time to 60 minutes. Substituting 50%CPA and 50%MK by weight of binder (precursor) in geopolymer mortar is the optimum amount to achieve the good compressive strength and enhanced slant shear bond strength. Grooved surface treatment method gave the highest slant shear bond strength with 7.31 N/mm² value. The interfacial transition zone through Scanning Electron Microstructure (SEM) showed that geopolymer made from CPA and MK is chemically bonded to the concrete substrate. The increase in Ca⁺⁺ ion balanced the negative charge of Al³⁺ ions, which lead to a dense interface zone. All the test results indicate that there is potential for the concrete industry to utilize alkaline activated blended Cassava Peel Ash and Metakaolin as an alternative to Portland cement in repair of concrete.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Growth in human population over the last fifty years has doubled from 3 to 6 billion and it is expected to increase in years to come. It is estimated that by year 2050, more than 85 percent of the world's population would live in urban areas. (Magnani *et al.*, (2015). To serve the needs and changes, large amounts of materials are needed for the construction of houses, office buildings, roads required for decent living (Ngab, 2002). Concrete as a construction material has the largest production of all man made materials. The worldwide consumption of this material is of the order of ten billion tonnes per year, next only to total consumption of water (Nagaraj, 2002). One of the constituent materials in making concrete is cement. Knowing that the raw material from earth sources was scarce and the demand for cement is increasing as a result of economic and population growth, the effort to find alternative materials which must be of both inexpensive and require very little energy to produce has to be undertaken. Thus, it is a current trend nowadays to use by-products or waste materials to partially replace cement in making concrete (Kartini *et al.*, 2006).

Cement manufacturing industry is one of the carbondioxide (CO₂) emitting sources besides deforestation and burning of fossil fuels. Global warming is caused by the emission of greenhouse gases, such as CO₂, to the atmosphere. Among the greenhouse gases, CO₂ contributes about 65% of global warming (Magnani *et al.*, (2015). The global cement industry contributes about 7% of greenhouse gas emission to the earth's atmosphere. In order to address environmental effects associated with cement manufacturing, there is a need to develop alternative binders to make mortar

and concrete. Consequently, extensive research is ongoing into the use of cement replacements, using many waste materials and industrial by products (Kartini *et al.*, 2006). To reduce the impact on the environment due to industrial and agricultural waste products such as Rice Husk Ash (RHA) and coconut fibers (COIR) which are the waste products of paddy and agricultural industry (Domke, 2012 ; Kartini *et al.*, 2006). Use of these waste products in mortar and concrete not only improves the strength of concrete but also leads to the proper disposal of these materials, resulting in reducing the impact of these materials on environment. Due to fast growth in population, the amount and type of waste materials have increased accordingly. Many of the non-decaying waste materials remain in the environment for hundreds, perhaps thousands of years. The non-decaying waste materials cause a waste disposal crisis, thereby contributing to the environmental problems. The environmental impact can be reduced by making more sustainable use of this waste. This is known as the Waste Hierarchy shown in Plate 1. The aim is to reduce, reuse, or recycle waste for the purpose of preventing the detrimental harmful effect, the latter being the preferred option of waste disposal (Magnani *et al.*, 2015).



Plate I: Waste Hierarchy
Source: Magnani *et al.*, (2015).

Considerable efforts has been taken Worldwide to utilize the industrial and agricultural waste and its by-product materials having high silica content as supplementary cementing materials or alternative materials to improve the properties of cement or replacement of cement in mortar and concrete (Domke, 2012).

Geopolymer materials can be obtained by recycling industrial by-products (e.g. fly ash and blast furnace slag), with no additional release of carbon dioxide (Duxson *et al.*, 2007). In addition, Geopolymer materials offer other advantages, such as high early strength development, improved chemical durability and excellent adhesion to other materials (Fernández-Jiménez *et al.*, 2006; Palomo *et al.*, 1999).

Declining infrastructure worldwide is requiring major repairs; the concrete industry is facing the concurring urgency of reducing their carbon emissions. Geopolymer materials represent a viable alternative to Ordinary Portland Cement (OPC) concrete in this regard. Geopolymer materials are produced from the alkaline activation of aluminosilicates, such as metakaolin, fly ash and blastfurnace slag, as well as from other silica and alumina rich minerals, residues and wastes (Juenger *et al.*, 2011; Shi *et al.*, 2011). The manufacture of those binders (e.g. metakaolin) requires lower temperature than OPC, with much less release of carbon dioxide; alternatively, compared to Portland cement.

The possibility of employing Geopolymer mortar as a more sustainable repair material compared to OPC concrete has been suggested by researchers in the last two decades (Davidovits, 2002; Pacheco-Torgal *et al.*, 2012) and the application of different types of Geopolymer materials for external reinforcement of reinforced concrete members was investigated (Menna *et al.*, 2013; Balaguru *et al.*, 1997). The

suitability of a material for repair relies upon several factors: foremost, bond strength and compatibility. Therefore, this present study focused mainly on the bonding strength properties of geopolymer concrete/mortar as a repair material incorporated binary blend of cassava peel ash (CPA) and metakaolin (MK) in shear strength bond.

1.2 Statement of the Research Problem

Repair is crucial in the rehabilitation or renewal of reinforced concrete (RC) structures. Although cement-based binders are usually employed in these repairs, the replacement of ordinary Portland cement (OPC) by Geopolymer binder is attractive when addressing the environmental concerns associated with the OPC production and its carbon footprint. Recently, there is a shift towards the use of geopolymer (GP) mortar in a wide range of repair applications. Test results by Zhang *et al.* (2010) confirmed the potential use of GP mortar as a protective coating for offshore concrete structures. Coppola *et al.* (2018) developed a slag based GP mortar to retrofit existing masonry and concrete buildings. Songpiriyakij *et al.* (2011) examined the potential of GP paste as a bonding agent in rebar embedded in a concrete substrate. It was observed that the GP paste developed high bonding strength, which was 24%–81% higher than the commercial epoxies. Ferone *et al.* (2013) utilized metakaolin (MK) based GP mortar for the external strengthening of reinforced concrete beams. Despite the intensive research undertaken on GP mortars for understanding the geopolymerization mechanism and optimizing the constituents for attaining enhanced strength (Fernández-Pereira *et al.*, 2018; Pires *et al.*, 2019; Huseien *et al.*, 2017), the appropriate selection of GP mortar is still under active research. The repair efficiency depends on the bond strength between the concrete substrate and the overlay repair

mortar. There is limited research on the performance of plain and agricultural waste based GP through the shear bond test.

1.3 Aim and Objectives of Research

The aim of this research is to determine the influence of cassava peel ash and metakaolin based geopolymer mortar on the bond strength of concrete with a view to determining its suitability and compatibility in retrofitting concrete. The objectives of the study are to:

- i. Characterise the properties of the pozzolan materials.
- ii. Determine the fresh properties of synthesized binary blend geopolymer mortar.
- iii. Compare the bonding properties of CPA-MK blend geopolymer mortar with normal CEM I concrete substrate.
- iv. Evaluate the morphological properties of the interface zone between geopolymer and CEM I mortar substrate.

1.4 Scope of Study

The research work was experimental in nature and centres on the feasibility of achieving a new geopolymer mortar with improved shear bond strength. This new binary blend geopolymer mortar can be achieved by combining MK and CPA in appropriate proportions. The series of tests procedures for determining this new geopolymer mortar was conducted based on the procedures of British Standards (BS) and American Society for Testing and Materials (ASTM).

1.5 Justification for the Research

This study anticipates to generate new information on the use of agricultural waste base geopolymer mortars by means of systematic methods of sample preparation from waste materials economically, appropriate and careful materials characterizations, and subsequent data analyses useful for the development of standard specifications for and its compatibility with old surface of existing concrete surface substrate. This generated knowledge is expected to contribute to the development of environmentally friendly and inexpensive geopolymer material for wide range of applications in the construction industry. This would be greatly beneficial for sustainable development of Nigeria, where wastes disposal problems towards the land filling can be avoided and minimized.

The outcome of the study is believed to provide the basis for further researches and better understanding of the bond behavior of geopolymer mortar obtainable from waste material supporting the concept of sustainability to concrete substrate.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Geopolymer Mortar

Geopolymers are inorganic materials that polycondense similar to organic polymers. The reaction of Al_2SiO_3 with alkali polysilicates produces an amorphous to semi-crystalline three-dimensional structure of polymeric sialate (Si-O-Al-O) bonds (Davidovits, 1994). The tetrahedral configuration of sialate, an abbreviation for alkali silicon-oxo-aluminate, is illustrated in Figure 2.1 with potassium, sodium, calcium or lithium being the alkali (Davidovits, 1975).

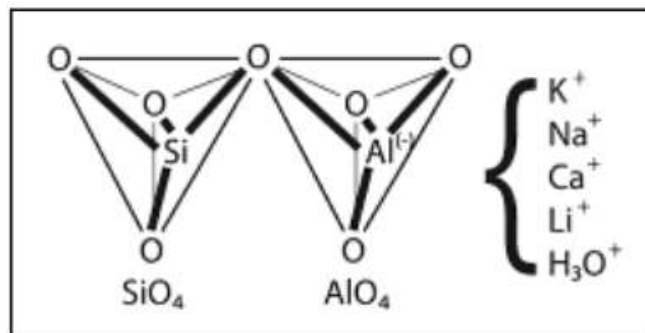


Figure 2.1: Tetrahedral Configuration of Sialate

Source: (Davidovits, 1975).

Through the action of hydroxide (OH^-) ions, the Al_2SiO_3 dissolves from the source material. Precursor ions then organize into monomers and polycondense to form polymeric structures (Hardjito *et al.*, (2005). Poly (sialates) have the following empirical formula (Davidovits, 2002): $\text{Mn} [-(\text{SiO}_2)_z - \text{AlO}_2]_n$, H_2O is released during the formation and curing of the geopolymer matrix, leaving behind discontinuous nano-pores that influence performance. Contrasting the hydration required for PC,

H₂O is not necessary for the chemical reaction to occur. The role of H₂O is to simply produce a mixture that is adequately workable (Hardjito *et al.*, 2005). Natural sources of Al₂SiO₃ include kaolinite, clays, micas, andalusite and spinel. However, source materials can also be byproducts of industrial processes, such as silica fume, slag and Fly Ash (FA). The geopolymerization of the source materials is generally accomplished through the use of an aqueous colloidal alkali polysilicate solution based on sodium that contains diverse forms of silica (Brykov, 2004; Leelathawornsuk, 2009). In contrast to geopolymer, the production of PC results from the calcination (thermal decomposition) of CaCO₂ and silico-aluminous materials, such as clay, shale or silica sand (Davidovits, 2002). Hydration of the resulting calcium silicate and calcium aluminate forms calcium silicate hydrate (C-S-H), calcium aluminate hydrate (C-A-H) and Ca(OH)₂. Formation of these compounds generates heat, causing thermal expansion and strength development (Taylor *et al.*, 2007).

2.2 Alkaline Activator

The alkali polysilicate solution is most commonly composed of NaOH and Na₂SiO₃ for geopolymers based on FA. It is also recommended for the NaOH component to be 25-100% NaOH with up to 75% H₂O and for the Na₂SiO₃ component to be 38-55% Na₂SiO₃ with 45-62% H₂O by mass and a SiO₂ : Na₂O ratio of approximately 2:1 to 3.22:1 (Silverstrim, 1997). The addition of Na₂SiO₃ allows the quantity of the mineral component to be reduced, and including it in the mixture is also advantageous for faster hardening of the binder. The benefit of incorporating NaOH into geopolymer

mixtures is its exothermic capacity as a strong chemical base to increase dissolution and polycondensation of the source material (Davidovits, 1975).

2.3 Applications of Geopolymer Mortar

The oldest geopolymer artifact is the Venus from Dolni Vestonice, a 27000 year old ceramic figurine. Clay was used as a natural source of Al_2SiO_3 and alkaline soluble salts, generating a chemical reaction when heated over a fire (Davidovits, 2002).

An investigation of the origin of geopolymer binders has also provided evidence indicating that the Great Pyramids of Giza were built using similar technology. The theory speculates that Egyptian workers mixed crushed limestone with H_2O from the Nile River for the required Al_2O_3 and SiO_2 . Locally available salts were then used to make the mixture alkaline. Opposing traditional engineering theories, the chemistry theory claims that the mixture was cast into wooden molds and cured in the desert heat to form pyramid stones. Similar conjectures have been made concerning antiquated structures in Italy and Germany (Davidovits, 1994). Due to its modern rediscovery, there is potential for the new binder to be used for a wide variety of applications (Davidovits, 2002). Geopolymers are being investigated in many scientific and industrial disciplines, including modern inorganic chemistry, physical chemistry, colloid chemistry, mineralogy, geology and other engineering process technologies. Bricks, ceramics and fire protection materials can be made from geopolymers with a Si : Al atomic ratio of 1, corresponding to a three-dimensional network of poly(sialate). A binder consisting of sialate-siloxo (Si-O-Al-O-Si-O) bonds, however, corresponds with a Si : Al atomic ratio of 2 and can be used for

In recent years, there has been exponential growth in the body of literature regarding geopolymer science (Geopolymer Institute, 2010). The worldwide increase in geopolymer research has provided a substantial amount of insight concerning the methods and materials required for the alkaline activation of FA. However, due to a lack of standard geopolymer preparation and testing procedures, there is large variation in research methodologies. The variation in practices parallels deviations found in the chemical composition of the source materials. Even though there are classifications of FA, inconsistencies in composition are found between FA from different power plants and even between batches of FA from the same source. For this reason, many investigations have focused solely on optimizing the quantity, constituents and concentration of the alkaline activator.

Other studies have investigated the effect of curing geopolymer specimens at elevated temperatures to accelerate polycondensation and hardening. In search of optimal source materials, some investigations have also examined differences between geopolymers based on Class C and Class F FA, while others have compared coarse and fine FA. However, limited research has been conducted with regards to the rheological behavior of geopolymers (Poulesquen *et al.*, 2011).

2.4.1 Alkaline activation

Hardjito *et al.*, (2005) presented the findings of an experimental programme regarding the development of GPC based on Class F FA from the Collie Power Station in Western Australia. Commercial grade NaOH pellets (97% purity) and Na₂SiO₃ solution (14.7% Na₂O, 29.4% SiO₂, 55.9% H₂O) were utilized as the alkaline activator. NaOH pellets were dissolved in H₂O to prepare the NaOH solution. Similar

to concrete based on PC, aggregates occupied 75-80% of the GPC mass. Three different aggregate combinations were investigated. In the primary experimental design, the ratio of Na_2SiO_3 : NaOH was manipulated from levels of 0.4 to 2.5, and the molarity of the NaOH solution varied from 8M to 16M. Most mixtures also contained naphthalene sulphonate super plasticizer in the range of 0% to 2% to improve workability.

Compressive strength was selected as the benchmark parameter due to its importance in the design of concrete structures, but other parameters were also considered. The preliminary lab work revealed that higher H_2O content created segregation, resulting in lower strength. However, if the Na_2SiO_3 and NaOH solutions were mixed together 1 day before adding them to the solid constituents, then segregation ceased. Specimens did not immediately harden at room temperature, having a handling time of at least 24 hours (Hardjito *et al.*, 2005).

The FA and aggregates were mixed in an 80L pan mixer for 3 minutes, and then the water, superplasticizer and alkaline activator were added and mixed for an additional 4 minutes. A conventional slump test was conducted to measure the workability of the fresh concrete. Specimens were cast in steel molds and compacted by 60 manual strokes per layer in 3 equal layers followed by 10 seconds on a vibration table. 100 mm x 200 mm cylindrical concrete specimens were cast for determination of compressive strength, and 150 mm x 300 mm specimens were cast for determination of splitting tensile strength. After being wrapped with vacuum bagging film and twist tie wire, specimens were either cured in an oven or steam chamber (Kotwal, 2015).

The experimental study indicated that a higher concentration of NaOH and a higher ratio of Na_2SiO_3 : NaOH resulted in stronger specimens. Additional mixtures were designed with a 2.5 Na_2SiO_3 : NaOH ratio, as the test results for this parameter were considered to be consistent and more economical. Strength also increased as an effect of raising the curing temperature. Additional specimens were prepared and cured at 140°F for periods ranging from 4 to 96 hours, and longer curing time resulted in higher strength. The initial setting time of the GPC could not be measured due to the lack of a suitable method; however, it was determined that the fresh mixture could remain in ambient conditions for up to 2 hours without losing workability or compressive strength. Super plasticizer volumes up to 2% improved workability without lowering compressive strength. Strength increased if specimens were left in ambient conditions for up to 3 days before curing at elevated temperatures. Similar to concrete based on PC, increasing the H_2O Na_2O molar ratio decreased strength but increased workability of the GPC. Results of the study also indicate that strength increased with longer mixing times (Hardjito *et al.*, 2005; Kotwal, 2015).

Compressive testing also indicated that the geopolymer matrix was stronger than the crushed granite. An analysis of the stress-strain relationship of the GPC revealed that it conformed to the prediction model. The strains at peak stress were 0.0024 to 0.0026, which are similar to concrete based on PC. The splitting tensile strength was determined to only be a fraction of the compressive strength, although it was higher than the values recommended by the prediction model. Mustafa *et al.* (2011) studied geopolymer based on Class F FA, to determine the effect of oxide molar ratios of SiO_2 : Al_2O_3 , H_2O content of the alkaline activator and the Na_2SiO_3 content for each

activator : FA ratio. NaOH pellets (97-99% purity) were dissolved in H₂O to make a 15M NaOH solution. One day prior to adding the activator to the solid constituent, technical grade Na₂SiO₃ solution (9.4% Na₂O, 30.1% SiO₂, 60.5% H₂O) was mixed with the NaOH solution.

The alkaline activator: FA ratio was manipulated from levels of 0.3 to 0.4 during the experiment. Prior to casting the geopolymer into 50 mm cube specimens, FA was mixed with the alkaline activator and 17% H₂O by mass for 15 minutes. Specimens were cured for 24 hours at room temperature then for 24 hours at 70°C in a furnace. The specimens were demolded after curing at elevated temperatures and aged for 7 days at room temperature. To prevent H₂O evaporation, the molds were sealed with plastic during storage, curing and aging (Hardjito *et al.*, 2005; Kotwal, 2015).

Test results indicated that the compressive strength of the geopolymer was highest, up to 846495kg/m², when the Na₂SiO₃ : NaOH ratio was 1.0. This value corresponded with a SiO₂ : Al₂O₃ ratio of 4.12 and a H₂O : Na₂O ratio of 11. Decreased strength resulted from varying the constituents less or more than the suitable value (Kotwal, 2015). Guo *et al.* (2010) investigated the use of Class C FA for a geopolymer binder. NaOH pellets (99.2% purity) and Na₂SiO₃ solution (9.1% Na₂O, 29.2% SiO₂, and 61.7% H₂O) were acquired. In the experimental design, the molar ratio of SiO₂: Na₂O in the mixed alkali activator was manipulated at levels of 1.0, 1.5 and 2.0. For each molar ratio, the content of the activator varied from 6% to 15% by the mass proportion of Na₂O: FA. The mass ratio of H₂O: FA remained at a constant level of 0.40, including the H₂O in the mixed alkaline activator. The geopolymer binder was mixed at approximately 22.8°C; however, the procedure for mixing was not

described. The fresh paste was then poured into 1" cube molds and covered with a vinyl sheet to prevent moisture loss and carbonation of the surface. Half of the specimens were cured at 22.8°C for 3, 7 and 28 days, and the other half were cured at elevated temperatures of 60°C to 90°F for 4, 8 and 24 hours. The optimum molar ratio of SiO₂ : Na₂O for improving compressive strength was 1.5, and the proper content of the activator was 10% by the mass proportion of Na₂O : 13 FA. Compressive strengths of 22,601 KN/m², 34,501 KN/m² and 59,301 KN/m² were measured for specimens cured at 22.8°C for 3, 7 and 28 days, respectively. The elevated curing temperature of 75°C produced the strongest specimens, and higher compressive strengths were measured as specimen age increased. Compressive strengths of 30,598 KN/m², 35,597KN/m² and 38,796 KN/m² were measured for specimens at 4, 8 and 24 hours, respectively, when the molar ratio and activator content were at the optimum levels.

2.4.2 Ambient curing versus heat curing

Vijai *et al.* (2010) studied the effect of curing conditions on the compressive strength of GPC based on Class F FA. NaOH flakes (98% purity) and Na₂SiO₃ solution (14.7% Na₂O, 29.4% SiO₂, and 55.9% H₂O) were used as the alkaline activator. The NaOH flakes were dissolved in H₂O to make an 8M NaOH solution, and the ratio of Na₂SiO₃ solution: NaOH solution was fixed at 2.5. A 0.4 ratio of activator: FA was also maintained. Aggregates composed 77% of the GPC mass, and 30% of the aggregate was river sand. Extra H₂O and super plasticizer were also added to the mixture to achieve adequate workability. The NaOH and Na₂SiO₃ solutions were mixed 1 day prior to combining them with the solid constituents. Aggregates and FA

were mixed in a pan mixer for 3 minutes, and then the alkaline activator was added and mixed for an additional 4 minutes. 6" cube specimens were cast and subjected to mechanical vibration. After 5 days, the specimens were demolded and cured in different conditions. Half of the specimens were left in ambient conditions, and the other half were cured at 140°F in an oven for 24 hours. The density of the hardened GPC specimens was found to be similar to concrete based on PC, ranging from 141 lb/ft³ to 150 lb/ft³. Only slight variations in density were measured due to the effect of age and curing type. Specimens that were cured in ambient conditions obtained a compressive strength of 3.89 N/mm² after 7 days and 17.7 N/mm² after 28 days. However, specimens cured in the oven reached strengths of 28.3 N/mm² after 7 days and 33.22 N/mm² after 28 days. Skvara *et al.* (2007) examined the properties of GPC based on FA. Concrete was prepared by mixing fine and coarse aggregate with FA and an alkaline activator. The aggregate gradation was in accordance with ISO 3310-1(2016) standards. The SiO₂: Na₂O molar ratio of the alkaline activator was manipulated by combining NaOH with soluble glass, and molar ratios ranging from 1 to 1.6 were considered. The ratio of the activator Na₂O: FA by mass varied in the range of 6% to 10%, and H₂O: FA ratios ranging from 0.30 to 0.40 were also used in the experiment. Ground slag, gypsum and limestone were added to the concrete in some cases. Manjunath *et al.* (2011) investigated the mechanical behavior of ambient cured GPM containing the following industrial byproducts: quarry dust, silica fume, ground granulated blast furnace slag and Class F FA. Commercial grade NaOH pellets (97-98% purity) and Na₂SiO₃ liquid were utilized as the alkaline activator. Variables manipulated for GPM composition included the binder: fine aggregate ratio (1:2, 1:2.5), FA: slag: silica fume ratio (60:40:0, 60:35:5, 50:50:0, 50:45:5, 40:60:0,

40:55:5), activator molarity (12M, 14M) and activator: binder ratio (0.4, 0.45). A constant ratio of 1:2 was maintained for NaOH: Na₂SiO₃. For each set of parameters, the binder components were first combined with the quarry dust, and then the activator solution was added and mixed until a homogeneous mortar resulted. 75 mm cube specimens were cast and compacted with a vibrator.

2.5 Kaolin

Metakaolin can be described as a dehydroxylated pozzolanic product derived from the high temperature firing of raw kaolin. Kaolin or kaolinite (Al₂Si₂O₅(OH)₄) is a clay mineral which contain high amount of layered tetrahedral silicon atom connected via oxygen to octahedral aluminum atom (Peterman *et al.*, 2010). ASTM C618 (2018) classifies metakaolin as a Class N (or natural) pozzolan. The Meta prefixes attached to kaolin connote change and the change that occurs in this context is the dehydroxylation. Dehydroxylation is the decomposition of kaolinite crystals to a partially disordered structure. Metakaolin is unique in that it is not the by-product of an industrial process nor is it entirely natural; it is derived from a naturally occurring mineral and is manufactured specifically for cementing applications. Unlike by-product pozzolans, which can have variable composition, MK is produced under carefully controlled conditions to refine its color, remove inert impurities, and tailor particle size (Ding, 2002). Dehydroxylation of kaolinite occurs at temperatures as low as 500–550°C, but the thermal treatment temperature of 800°C recommended in the original. Many advantages have been reported on the uses of metakaolin as raw material in the synthesis of geopolymer. Some of these advantages include; reduction of efflorescence (a whitish haze which is caused when a calcium hydroxide reacts

with carbon dioxide in the atmosphere), increase or boost compressive and flexural strengths, mitigate against chloride and other permeability, increase resistance to acid attack and durability of the geopolymer (Olawale, 2013). Kaolin can be found in abundance in many parts of Nigeria as shown in Table 2.2.

Table 2.2: Kaolinitic Clay Deposits in Nigeria

Region	State (Location)	Reserve (MillionTonnes)
North	Kaduna (Mararaban-Rido)	5.5
	Katsina (Kankara, Safana, Malumfashi, Musawa)	Very large
	Faskari, Ingawa, Dushin-Ma, Bakori)	Very large
	Kebbi (Ilo, Kaoje)	Very large
	Plateau (Major Portar, Nahuta)	8.0
	Borno (Maiduguri)	Large
	Kwara (Lafiagi)	Large
	Bauchi (Darazo, Jagalwa)	18
	Benue (Apa, Iga Okpava, Okpaga)	10
	Niger (Gubaji,Gbako)	very large
South	Anambra (Ogubulu)	4.2
	Enugu (Enugu)	50
	Oyo (Tede)	1.5
	Ogun (Onibode, Ibamaje, Miroko)	large

Source: RMRDC MCTS Survey Report, (2003); NMC (2003)

2.6 Cassava Peel Ash

Cassava is known to be a major source of carbohydrates with Africa being the largest centre of production. (Adesanya *et al.*, 2008) reported that cassava peel constitutes between 20-35% of the weight of tuber, especially in the case of hand peeling. Based on 20% estimate, about 6.8 million tonnes of cassava peel is generated annually and 12 million tonnes is expected to be produced in the year 2020. Indiscriminate disposal of cassava peels due to gross underutilization (Ayangade *et al.*, 2004; Olanipekun *et al.*, 2006), as well as lack of appropriate technology to recycle them is a major challenge, which results in environmental problem. These wastes would even be more problematic

in future with increased industrial production of cassava products such as cassava flour and garri. In the same vein, developing nations of the world are challenged with issues of managing domestic and agricultural wastes as a result of the attendant growth in population and increasing urbanization. Reuse of these wastes provide an attractive option that promotes savings and conservation of natural resources from further depletion hence creating a sustainable environment. Solid waste and its resource potential are being appraised for reuse (Ofuyatan & Olutoge 2014). Thus, there is need to search for alternative methods to recycle them (cassava peels). Cassava peels burnt at controlled or uncontrolled temperature showed that its ash possessed an appreciable quantity of silica and alumina for use as pozzolan in concrete (Olonade & Mohammed, 2019). According to Salau & Olonade (2011), the pozzolanic potential of cassava peel ash (CPA) and their results showed that cassava peel ash possesses pozzolanic reactivity when it is calcined at 700⁰C for 90 minutes. At these conditions, CPA contained more than 70 per cent of combined silica, alumina and ferric oxide. Hence, in accordance with the chemical composition of Pozzolans specified by ASTM C618 (2018).

2.7 Interfacial Stresses

Stresses on the bond interface of repairs in the field can be affected by factors such as those listed as; plastic and drying shrinkage strains in the repair material, heat generation from early heat of hydration or polymer reaction thermal stresses (including thermal shock when hot repair material is exposed to cold ambient temperatures), time dependent volume changes, such as drying shrinkage (or expansion in shrinkage compensated Concretes), autogenous shrinkage, carbonation shrinkage and creep, dead loads and changing live loads and dynamic loads, Thermal stresses from diurnal or seasonal

temperature changes, or external heat sources, Frost build-up or salt crystallization pressures, other factors such as impact loads or changes in moisture gradient in the repaired system (Zhifu, 2011).

2.8 Repair Techniques

A few methods for fixing or potentially reinforcing structures include adding new Concrete to a current substantial substrate. To improve the security strength, it is not unexpected to build the unpleasantness of the substrate surface. The surface harshness, the utilization of a holding specialist and the dampness substance of the substrate can have a huge impact in the security strength of the interface and disappointment method of composite substantial individuals with layers cast at various ages (Zhifu, 2011).

Since composite substantial individuals are projected at various ages, various Concretes are every now and again received. With a similar blend plan, contrasts are acquired in the compressive strength and in the Young's modulus. For this situation, the most fragile substantial layer controls the disappointment of composite individuals. Moreover, differential firmness because of various Young's modulus at each layer likewise influences the conduct of the composite individuals since extra burdens are prompted at the interface (Zhifu, 2011, Tayeh *et al.*, 2012).

2.8.1 Surface roughness

Readiness strategies, for example, wire-brushing, sandblasting, shot-impacting, chipping and hydro destruction are often times used to eliminate the shallow layer. Moreover, to the unpleasantness expanding technique, a holding specialist can be utilized to improve the bond strength. For this situation, epoxy-based tars are the most regularly received in bond new/solidified to solidified substantial parts however the subsequent advantages are not

broadly acknowledged by specialists. Some propose that a satisfactory security can be just accomplished by joining the utilization of holding specialists with an appropriate strategy to expand the substrate harshness basically when the substrate presents a smooth surface. The examples with the substrate surface treated with sand-impacting showed the most noteworthy upsides of security strength in shear (14.13 N/mm^2) and the least upsides of variety coefficient (8.56%) (Zhifu, 2011, Ogunbode *et al.*, 2021). Recently, many researchers tried different types of surface roughness and identify the best method in term of practicality and strength (Nath & sarka, 2015; Islam *et al.*, 2014). This is because the bond strength depends not only on the characteristics of the repair material but also on the surface roughness of the concrete substrate. Bond strength between the repair materials and substrate depends on the surface treatment of the substrate, as the surface treatment increases the bond strength increases (Tayeh *et al.*, 2012). Geopolymer possess very high bond strength however, some researchers believed that it is not affected by increasing the surface roughness of the concrete substrate (Pacheco-Torgal *et al.*, 2008). The surface substrate that is rich in calcium hydroxide but lack exposed coarse aggregates which could contribute to improve bond strength due to silica dissolution from the aggregate surface. However, it is believed that surface treatment have a significant impact to the bonding between repair material (CPA/MK geopolymer mortar) and concrete substrate.

2.8.2 Bonding agents

At the point when the substrate is immersed or presents high dampness content, even with its surface dry the impact of the surface planning is less huge. In these conditions the utilization of a holding specialist is beneficial yet in addition less huge in contrast with similar conditions however with a dry substrate. Besides, the impact of the surface

harshness gives off an impression of being more huge when concrete mortars or polymer adjusted concrete mortars are utilized, since when epoxy tars are embraced disappointments don't habitually happen at the interface. Other than unpleasantness and holding specialists, the impact of boundaries like temperature, and specifically the impact of cyclic varieties ought to be assessed for every particular circumstance since they can handle the conduct of the interface (Mohammadi, *et al.*, 2014).

2.8.3 Curing of concrete

Current plan codes for substantial designs don't expressly introduce arrangements for the relieving methodology of composite individuals cast at various ages and subsequently the impact of differential shrinkage is frequently dismissed. This is a vital boundary since various Concretes with various relieving conditions for sure exist in composite individuals. It is regular prescribed to begin relieving following the cast of the additional substantial broadening it for in any event 3–7 days to improve the bond strength. Boundaries like relative stickiness and temperature, just as the openness to wind, downpour and sun powered radiation should be thought of (Pedro & Eduardo, 2010).

2.9 Test Method

A few tests are accessible to quantify the bond strength, yet just little data is accessible on correlation of these different tests' strategies and the subsequent security strength values. There is a need to think about various tests for estimating bond strength and to build up a relationship among the qualities acquired from each test (Pedro & Eduardo, 2010).

The bond strength predominantly relies upon attachment in interface, grinding, total interlock, and time subordinate elements. Every one of these principle factors, thus relies upon different factors. Attachment to interface relies upon holding specialist, material compaction, cleanness and dampness substance of fix surface, example age, and unpleasantness of interface surface (Pedro & Eduardo, 2010).

Grinding and total interlock on interface rely upon boundaries like total size, total shape, and surface planning. Notwithstanding the above factors, the deliberate security strength is exceptionally reliant upon the test technique utilized. Size and calculation of example and the condition of weight on the contact surface are very reliant upon the picked test technique. It is noticed that specific standard tests have been created for explicit applications and condition of pressure. There are two issues that should be tended to. To start with, what kinds of tests are suitable for assessing the bond strength for the condition of pressure that is usually found in structures, i.e., shear pressure brought about by stacking and time subordinate components. Second, what relationship exists between the consequences of various test techniques (Pedro & Eduardo, 2010). The existing tests to determine the bond between concrete substrate and repair material can be divided into several categories.

2.9.1 Bond under tension stress

The first category of tests measures the bond under tension stress. Pull-off, direct tension and splitting are the main tests under this category.

2.9.1.1 *Pull-off test*

The draw off test is a pressure test and has been decided for two reasons (1) to assess the bond strength in strain of the interface and (2) it very well may be done in situ.

The embraced math for the draw off examples was a 0.20 m block with the interface line at the centre. A centre of 75 mm breadth was penetrated into the additional substantial and expanding 15 mm past the interface into the substrate. A round steel plate was fortified, with an epoxy pitch, to the outside of the centre. A strain power was applied to the circle, with a business gadget at a consistent pace of $0.05 \text{ N/mm}^2/\text{s}$, until disappointment happened (Pedro & Eduardo, 2010).

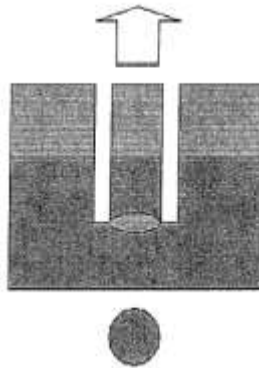


Plate II: Pull-off bond test
Source: Pedro & Eduardo (2010)

2.9.1.2 Direct tension test

In the immediate strain test, the tractable power is communicated to the substantial example either by stuck metal or by unique holds. An exceptionally cautious arrangement of the example in the pivot of stacking is fundamental. Indeed, even an exceptionally limited quantity of misalignment may present whimsies that was cause enormous disperse in test results. Playing out a decent strain test is troublesome and tedious. Nonetheless, an as of late proposed variety of the immediate pressure test, alluded to as pull off test, is simpler to do and can create great outcomes. Plate III Presents a pictorial representation of direct tensile bond test (Pedro & Eduardo, 2010).



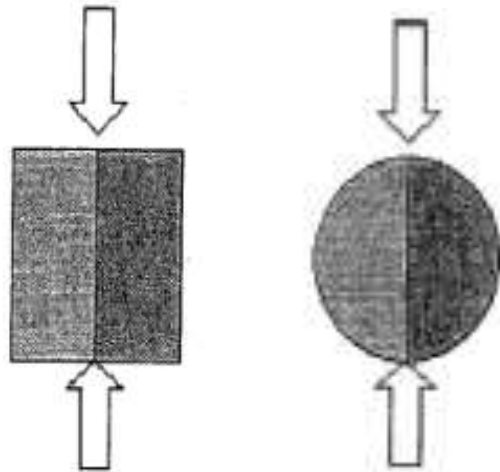
Plate III: Direct tension bond test
Source: Pedro & Eduardo (2010)

2.9.1.3 *Indirect tension test*

Roundabout strain tests incorporate the flexural test and the parting test. The flexural test offers low proficiency (the space of the fortified surface exposed to stacking is little contrasted with the example volume). For such tests, just an exceptionally little piece of the reinforced plane is exposed to the most extreme burdens. Parting test is more effective around there. In the parting test, a crystal with roundabout or square cross-area is put under longitudinal compressive stacking. Strain stresses cause disappointment in a plane going through upper and lower tomahawks of stacking and split the example into equal parts. The parting elasticity of Concrete is viewed as a sign of its rigidity. The test strategy is easy to perform and utilizes a similar barrel shaped example and test machine as a standard pressure test. The parting tractable test according to ASTM C496 (2016), as a roundabout ductile test, was led to assess the security strength between the NC substrate and Concrete (Pedro & Eduardo, 2010).

A crystal with roundabout or square cross-area is set under longitudinal compressive stacking. Strain stresses cause disappointment in a plane going through upper and lower tomahawks of stacking and split the example into equal parts. The parting

elasticity of Concrete is viewed as a sign of its rigidity. The test strategy is directed in congruity with ASTM C 496 (2016), as a circuitous pliable test, to assess the security strength between the new concrete and substantial substrate as represented in Plate II..



(a) **Square splitting**

(b) **Cylinder splitting**

Plate IV: Indirect bond test under state of tension stress
Source: Pedro & Eduardo (2010)

2.9.2 Bond under shear stresses

The second category of tests measures the bond under shear stresses and is called direct shear methods. Several tests fall under this category, including L-shaped, mono surface shear, etc.

2.9.2.1 *Direct shear test*

Most of the time, the bond surface for an immediate shear test is really exposed to shear pressure and a little twisting pressure. At the point when a steel plate is utilized to send the shear power along the bond line, some pressure fixation at the edge of the

holding plane is incited. More modest pressure focus prompts more modest dissipate in test results.

2.9.3 Bond strength under a state of stress

The third category measures the bond strength under a state of stress that combines shear and compression. All slant shear tests mentioned previously fall under this category.

2.9.3.1 Slant shear test

The inclination shear test utilizes a square crystal or a barrel shaped example made of two indistinguishable parts reinforced at 30° or 45° and tried under hub pressure and during stacking; the interface surface is under pressure. The inclination shear test according to ASTM C882/C882M (2020) has become the most generally acknowledged test and has been embraced by various worldwide codes as a test for assessing the security strength of resinous fix materials to substantial substrates. In any case, there is no broad understanding among scientists with regards to the fittingness of this test for non-resinous materials.

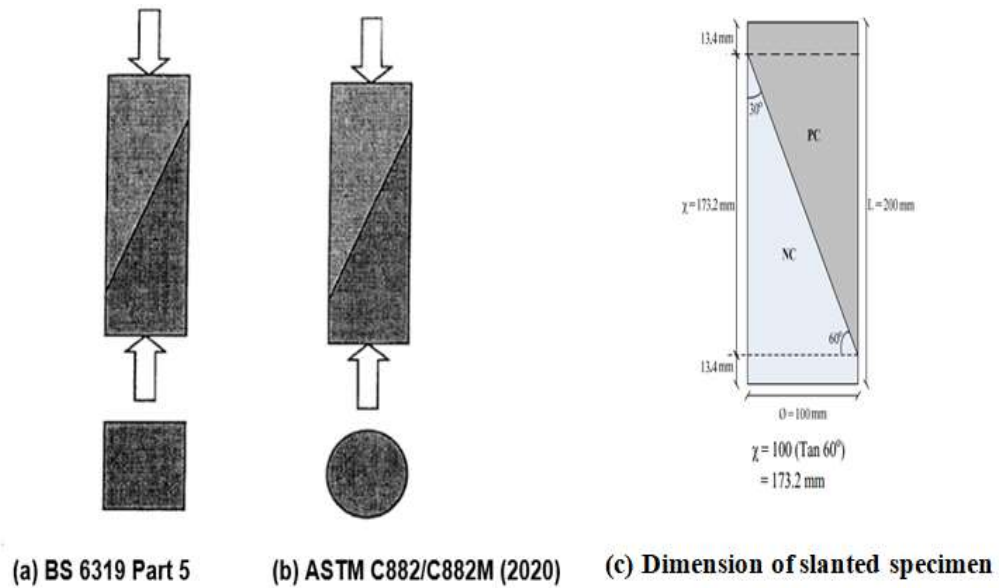


Plate V: Slant shear test using either (a) square prism or (b) cylindrical specimen bonded at 30° or 45° and tested under axial compression loading, (c) Dimension of slanted specimen

Source: Pedro & Eduardo, (2010)

2.10 Minimum Acceptable Bond Strength Range

According to American Concrete Institute (ACI) Guide - Concrete Repair Guide (Chynoweth *et al.*, 1996), materials used in concrete repair work shall have a specified minimum acceptable bond strength based on the slant shear strength as presented in Table 2.3.

Table 2.3: Minimum Acceptable Bond Strength Range

Days	Bond Strength (S) N/mm ²
1	2.76 - 6.9
7	6.9 - 12.41
28	12.41 - 20.68

Source: Chynoweth *et al.* (1996)

This guide is useful in the selection of appropriate type of repair materials for rehabilitating deteriorated concrete structures.

2.11 Summary of Literature Reviewed

Based on the conclusions found within the literature regarding the synthesis of geopolymers, the following claims can be made:

1. Increasing the NaOH and Na₂SiO₃ content reduces the flow of fresh geopolymer. Workability can be improved, however, with the addition of extra H₂O.
2. Excessive OH⁻ ions accelerate dissolution but decrease polycondensation, causing the binder to precipitate early and lose strength.
3. Excessive H₂O content creates segregation between the constituents and lowers the strength of the final Concrete.
4. At higher rate of mixing leads to an increased amount of air content, which remains enclosed in the fresh concrete due to its low mobility. Additives that contain CaO reduce porosity as the geopolymer phase coexists with the C-S-H matrix.
5. GPC containing high amounts of FA has increased levels of static and dynamic viscosity and, therefore, requires higher compaction energy.
6. Decreased strength results from varying the constituents of the alkaline activator less or more than the suitable value.
7. Strength increases as an effect of age, longer mixing time and raising the curing temperature.
8. The density of GPC is similar to concrete based on PC, varying only slightly due to the effect of age and curing type.

9. Fine FA is much more effective for synthesizing high strength geopolymers, providing increased surface area, high workability and excellent volumetric stability.

10. The mix design parameters as well as the fineness, crystallographic distribution and chemical composition of the FA dictate the mechanical behavior of geopolymers.

Hence, this study hereby aimed at filling the existing gap on the impact of binary blend of cassava peel ash (CPA) and metakaolin (MK) on the bonding shear strength of concrete/mortar as there is limited studies on the incorporation of CPA as an agro-waste material blended with MK as solid geopolymer repair materials.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The material that was used to achieve the aim and objectives of the study is described in detail below. The materials used in this research are; Cement, Metakaolin, Cassava Peel Ash (CPA), fine aggregate, coarse aggregate, Alkaline solution (Sodium Silicate (Na_2SiO_3) & Sodium Hydroxide (NaOH)), water and superplasticizer.

3.1.1 Alkaline solution

A combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) was used as the alkaline activator. The process is described as follows:

3.1.1.1 Sodium hydroxide (NaOH)

NaOH which is in pellet or flake form with 97%- 98% purity was obtained from a supplier and dissolved in tap water to make a solution. For this work, 9 Molar concentrations was used as the molarity for the mix. This indicates that the molarity was multiplied by the molecular weight of NaOH (40). This means ($9 \times 40 = 360$) which gave the quantity in grams of NaOH solids per litre of water.

3.1.1.2 Sodium silicate (Na_2SiO_3)

The sodium silicate solution ($\text{Na}_2\text{O} = 14.0\%$, $\text{SiO}_3 = 27.0\%$, and $\text{H}_2\text{O} = 59\%$ by mass) that was used for this work was purchased from a chemical supplier in Keteren Gwarri, Minna, Niger State.

3.1.2 Metakaolin

Metakaolin (MK) that was used for this research was obtained from kaolin, sourced from Alkaleri Local Government of Bauchi State, Nigeria. A bag of kaolin was air dried in Minna Niger State, then pounded to pellets using mortar and pestle. It was calcined to a temperature of 750°C for about 4hours. It was grinded using grinding machine and sieved with 75 micron sieve in the Department of Building Laboratory, Federal university of Technology, Minna. Chemical tests was carried out on the MK samples to determine the chemical compositions.

3.1.3 Cassava peel ash

Cassava peels (CPA) was collected using polythene bags from Doko, in Lavun Local Government Area of Niger State where Cassava flakes are locally produced. Twenty bags of the Cassava peels was first dried in open air and burnt to ash in close air in Doko after which it was sieved using 75 micron sieve and then calcined at a temperature of 500°C for 2hours then it was sieved again using same size of the sieve for final use. Chemical tests were carried out on the CPA sample to determine the chemical compositions.

3.1.4 Aggregates

The aggregates used in the experiment where fine aggregate and the coarse aggregate. The coarse aggregate was used in the production of the substrate concrete sample.

3.1.4.1 Fine aggregates

Fine aggregate river sand was obtained within Minna. It was kept in the SSD condition prior to use in the laboratory of the Department of Building, Federal

University of Technology Minna. Sieve analysis in accordance with BS EN 196-1 (2016) was carried out to determine the particle size using different sieve sizes and also remove impurities and bigger size aggregates.

3.1.4.2 Coarse aggregate

Air-dried crushed granite of 10 mm maximum size with specific gravity of 2.7 and water absorption of 0.5% was used as coarse aggregate in all the mixes. Care was taken to ensure that the aggregate is free from organic matters.

3.1.5 Cement

CEM 1 (42.5N) Portland Cement was used for this research as binders for the concrete substrate specimen. It satisfy the minimum requirement as provided in BS 12(1996). The CEM 1 Portland Cement was Dangote brand of cement from obajana plant conforming to the BS EN 197-1 (2000). The cement was acquire from a merchant store in Minna and kept in air tight container for use.

3.1.6 Water

Portable water fit for drinking supplied to the concrete laboratory was used throughout the study in mixing and dissolving the alkaline activator and also used for the production of the GPM overlay and concrete substrate specimens.

3.1.7 Admixture

A water reducing admixture was used in order to achieve a target flow. Super plasticizer (SP) of trade name CONPLAST SP 430 conforming to ASTM C494 (2019) requirements was used as a water reducing admixture to enhance the workability of mortar. The quantity of the super plasticizer used was 4% by the mass

of the binder. Owing to the high viscosity, the super plasticizer was dissolved as part of mixing water before adding to the fresh mortar during the mixing process.

3.2 Methods of Manufacturing Geopolymer Mortar

Manufacture of test specimen and the test procedure for evaluating fresh and hardened state properties and the standards adopted in conducting various test on strength and slant shear bond strength of the Geopolymer mortar (overlay) and the Portland cement concrete (PCC) (substrate) are presented and discussed. PCC (substrate) and GPM (overlay) that thus consistently meet the requirements of workability (flowability) and strength (shear bond strength) are only achieved through the application of stringent requirements on selection of material. In this study, targeted strength of the test sample was achieved through a well-considered material proportion design and also through the use of quality materials in the sample production. A mix proportion which is suitable for the production of PCC and GPM were determined through the preliminary test. The fresh properties test involves the testing for initial and final setting time, soundness and flowability of the GPM. Details of how this test are conducted are explained in the later subsections

3.2.1 Characterizations of the constituent materials

The properties test that was carried out on the constituent materials includes the physical and chemical properties of the constituent materials use for the research.

3.2.1.1 Physical properties

The physical properties of the materials used in the production of the Geopolymer mortar are conducted on the constituent materials like Fine aggregate, Coarse

aggregate, Precursors (CPA and MK) for this experiment are particle size distribution, (size analysis), density test and specific gravity test.

i. Particle Size Distribution

The particle size distribution for MK, CPA and fine aggregate was carried out using sieve analysis as described in accordance with BS EN 933-1 (2007). This was done to determine the grading of the aggregates.

ii. Specific Gravity

The specific gravity (Gs) of metakaolin, CPA, and fine aggregate was determined by using pycnometer method in accordance to BS EN 1097-2 (2003). The apparatus used include density bottle and stopper, funnel, spatula and weighing balance. The relationship used to find the specific gravity is given in Equation 3.1:

$$\text{Specific Gravity} = \frac{W_2 - W_1}{(W_4 - W_1) - (W_3 - W_2)} \quad (3.1)$$

iii. Bulk Density

Bulk Density was determined in accordance with BS 812-2 (1995) for the fine aggregates use for this research. The relation below is used to determine the bulk density of the sample: Equation 3.1 was used to calculate the samples bulk density.

$$D = \frac{M}{V} \quad (3.2)$$

D = Density of the aggregate specimen in Kg/m³

M = Mass of the aggregate specimen in Kg

V = Volume of the aggregate specimen in m³

iv. Density

In accordance to the ASTM C188 (1995), the density of ash such as CPA and MK used in this experiment was carried out using weight of compacted and uncompact samples.

3.2.1.2 Chemical properties

Chemical analysis of the precursors (CPA and MK) was carried out using XRF test to determine the oxide composition such as Silicon Oxide (SiO_2), Aluminum Oxide (Al_2O_3), Iron Trioxide (Fe_2O_3) and others. In order to investigate if they are in line with the ASTM C618 (2018) classes of pozzolana. The ASTM standard stipulates that for any material to be used as pozzolana, it should fall within the following classes; Class N, Class F or Class C.

3.3 Fresh Properties of Mortar

In concrete technology, the workability of concrete is measured by the experiment named slump test. However, for mortars, one of the flow table or the mini-slump cone was used in the fresh properties determination. Also the consistency of water test, initial and final setting time, soundness test is also determine for the CPA-MK geopolymer mortar.

3.3.1 Soundness of GPM

Accordance to BS EN 196-3(2016) the soundness test is a measure of the binder's quality in terms of expansion effect. Using a Le-Chatelier mould, this test was done at a temperature of $25 \pm 2^\circ\text{C}$ and humidity of $65 \pm 5\%$. It was completed following the consistency test (p) to determine the amount of water needed to make a standard paste. To make the paste, multiply 0.78 by the amount of water required to get a paste

with a standard consistency (0.78). After being oiled, the mould was placed on an oiled glass sheet and the binder pasted was poured to fill up before covering it with oiled glass sheet. The sample was put in water at temperature of $27 \pm 2^{\circ}\text{C}$ and kept for one day. The sample was taken from the water bath and the distance between indicator points was measured to the nearest 0.5mm (L1). The sample was re-immersed in the water bath, and the water bath's temperature was raised to boiling in 25 to 30 minutes for three hours, the water bath's temperature was lowered to room temperature then, the sample was taken out of the water bath. The results of measuring the distance between the two indication points was to the nearest 0.5mm (L2).

As a result, the following was used to determine the soundness test:

Soundness (expansion) of cement paste = $L1 - L2$

Where $L1$ = Value obtained after water immersion for 24 hours at $27 \pm 2^{\circ}\text{C}$

$L2$ = Value obtained after 3 hours immersion in water at boiling temperature.

Soundness value was computed from the mean of duplicate samples to the nearest 0.5mm

3.3.2 Setting time of GPM

This section elaborates on the method to evaluate the initial and final setting time of the mortar. The test method is adopted in measuring the setting time and consistency of GPM is called the vicat test method. This test is done in concomitant with ASTM C191 (2020). The vital tools for the vicat test are:

- (a) Standard Vicat apparatus which is a simple penetrometer with a variable sliding weight that can be attached to a needle

(b) Standard Vicat needles such as initial set needle, which is a not blunt ended cylinder needle of diameter 1.13 mm, a final set needle which is a cylindrical blunt ended shape with a hollowed metal collar with a diameter of 5 mm as presented in Plate II(b)

(c) Cone that acts as the mortar container and should be located in the apparatus with the proper needle attached.

(d) Glass plate is located under the mold to provide a smooth surface under the cement paste (mortar).



(a) Schematic diagram of a vicat apparatus



Initial Setting time test

Plate VI: Initial Setting time and Final settingtime test using the vicat apparatus

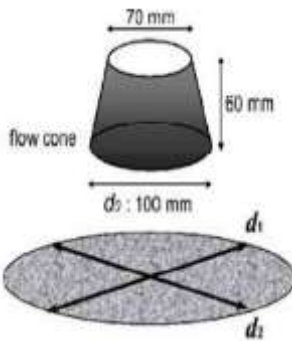
As mentioned in ASTM C191 (2020), the initial setting time is called to the duration from when the water becomes adjacent to the cement (binder) in the mixer until the time when the Vicat initial set needle penetrates 5 ± 1 mm to the mortar. To measure this time, turn the timer on while adding the water during the mixing process. When the mortar is thoroughly mixed, pour it into the Vicat mold and locate it under the

needle fitted on the apparatus. Next, calibrate the needle to gently touch the top surface of the mortar in the mold. Then release the needle and measure the penetration depth. Pull up the needle, recalibrate to the surface over the top surface, and repeat the procedure to reach the desired penetration mentioned above and read the time from a timer. The final setting time is called to the duration from when the water becomes adjacent to the cement (binder) in the mixer up until the time when the tip of the final set needle just marks the top surface of mortar but does not penetrate it. In order to measure this time, repeat the procedure above but with the proper needle (final set needle) and read the time at desired penetration again.

3.3.3 Mini-slump test for flowability of GPM

According to ASTM C143 (2020), the procedure for the mini-slump test used in the study is illustrated in this section. First, the Heidelberg measurement board or the base plate is place on a flat horizontal surface free from external vibrational shocks. The top surface of the base plate was checked for horizontality using a spirit level. The cone is placed centrally within the circle on the table (Plate I (a) shows the standard cone dimensions). Then, the cone was filled with GP fresh mortar immediately after mixing in a single operation without any mechanical compaction. The cone was allowed to stand not more than thirty seconds and, during this time, all spilled mortar was removed from the baseplate. The cone was lifted vertically in a single movement. In the end, without disturbing the mortar or the table, the diameter of the flow in two perpendicular dimensions was measured and the average was calculated. Plate I presents the operation procedure of the flowability test. Equation 3 illustrated the formula used in calculating the flow diameter

$$\text{Flow diameter} = \frac{(D_1 + D_2)}{2} \quad (3.3)$$



(a) Schematic diagram of a Mini slump test cone and diameters



(b) A geopolymer mix



(c) cone was lifted vertically in a single movement



(d) the diameter of the flow in two perpendicular dimensions was measured

Plate VII: Flowability test for Geopolymer Mortar

3.3 Production of GPM (overlay) and Portland cement concrete (substrate)

Specimens

The GPM (overlay) and Portland cement concrete (PCC) (substrate) specimen are produced following the mix design presented below and tested for compressive strength, tensile strength and slant shear bond strength.

3.3.1 Production of GPM (overlay) and Portland cement concrete (substrate) specimen

3.3.1.1 Mix design

Currently, no standard mix design is available for the production of Geopolymer (Huseien *et al.*, 2017). This means that the mix design for the production of Geopolymer is based on trial and error. For this reason, the method adopted by Huseien *et al.* (2017), in designing Fly Ash (FA), Granulated Blast Furnace Slag (GBFA) and Palm Oil Fuel Ash (POFA) mortar and concrete was used to design of CPA and MK based geopolymer mortar. Preparation of alkaline solution a combination of NaOH and Na₂SiO₃ was used as the alkaline solution in this research. For the purpose of this research, Molarity of sodium hydroxide was 9M and the ratio of sodium silicate to sodium hydroxide was 2.5. The various molar concentrations of NaOH pellet was dissolved in water to make a solution. The method that was used for mixing the Geopolymer specimens was similar to what was used when producing portland cement concrete (control). MK and CPA substituted at three (3) varying ratios 0, 50 and 100% and the aggregates (fine aggregate and coarse aggregate) was mix together thoroughly, after which the alkaline solution was added. After mixing, the fresh geopolymer concrete was cast into 100 mm x 100 mm x 100 mm iron mould, it was then left for 24hours (called resting period) before taken to the oven for curing at a temperature of 60°C for 24 hours.

The water cement ratio that satisfied the design mix for concrete made with CEM 1 for the substrate specimen and for comparison of strength with the Geopolymer

mortar sample was conducted using the 1:4 ratio. Both tests were conducted under the influence of water reducing admixture at constant quantity.

3.4 Tests on hardened of specimens

The tests to be carried out on hardened GPM (overlay) and Portland cement concrete (substrate) specimens include the following:

- (a) Compressive strength (ASTM C109/C109M, 2020)
- (b) Slant shear bond strength (ASTM C109 C109M, 2020)

3.3.1.2 Compressive strength

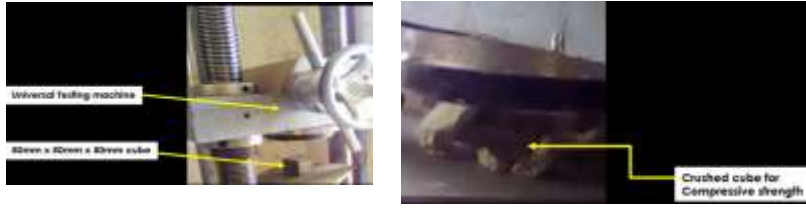
In order to evaluate the compressive strength of CPA-MK geopolymer mortar, a cubic specimen of 50 mm x 50 mm x 50 mm was used. The specimens were tested for ages of 3, 7, 28 and 56 days. According to ASTM C109/C109M (2020), the load was applied by a 2000KN hydraulic compression testing machine with 0.5N/mm²/sec of loading speed. A constant loading rate was selected and applied until failure of the specimen. Plate VIII shows the 50 mm mould, 50 mm mortar sample and testing of the 50 mm cube mortar sample and the failure pattern of the GPM cube that was used in the experiment.



(a) Steel Cubic mould containing fresh mortar



(b) GPM and PCM cubic Specimen



(c) Cube testing under compressive load

(d) GPM and PCC Cube failure pattern

Plate VIII: Compressive Strength test

3.3.1.3 Slant shear test

For half slant-shear specimen, the hardened CEM 1 cement concrete (substrate) also called Portland cement concrete (PCC) was diagonally slanted at 30° angle from vertical (Plate X). According to ASTM 882/C882M (2020), the bond angle of 30° recommended represents the failure stress corresponding to a smooth surface is close to the minimum stress. The dimension of slanted specimen is presented in Plate IX. Step-by-step preparation of half slanted specimen and bond test under uniaxial load are given in Plate X.

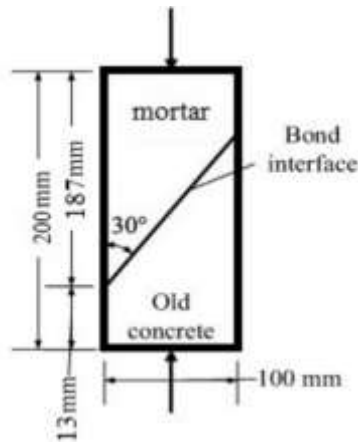


Plate IX: Slant shear specimen dimensions

Step 1

Polystyrene was cut and formed in cylindrical shape with diameter of 100 mm and length of 200 mm. Then, it was cut to half following the half slanted dimension (see Plate IX)



Step 2

Half slanted polystyrene was placed into the cylinder mould and fresh concrete was poured into the mould.



Step 3

Hardened CEM 1 PC (substrate) was water cured for 28 days. After the specimen was cured, the slanted area of hardened normal concrete was smooth roughed. This process was prepared to provide critical slanted area.



Step 4

Hardened CEM 1 PC (substrate) was placed again into the cylindrical mould. Then, fresh geopolymer mortar mix was poured into the mould, over the different surface treatment of the substrates. (Groove, smooth and mesh surface)



Step 5

A total of 72 specimens (Three specimens per mix type) were employed for slant-shear test. The test was conducted using compression testing machine (Specimen under load). The failure mode that is the collapsed pattern of the slant shear specimen is presented in the plates.



Plate X: Step of specimen preparation and testing procedure of slant-shear test

The vertical failure load was used to quantify the inclination shear test and in deciding the interface surface under pressure. The inclination shear test cylinder sample size for the geopolymer mortar is 100 mm diameter by 200 mm height. This is in agreement to ASTM 882/C882M (2020) as illustrated in Plate V (b & c), Chapter 2, Section 2.93.

There are various kinds of tests to evaluate the bond strength of mortars and concrete; however, to measure and compare the bond strength of each mix design in this research, the slant shear test was conducted in which a combination of both compression and shear stress was applied to the interface between the mortar and concrete. The specimens for slant shear test consist of two layers, a bottom substrate layer which is made of PC concrete and the same for all specimens, and a layer of mortar made of the mentioned mix designs (MK/CPA). The interface between these two layers makes an angle of 60° with the horizon and 30° with the vertical. Plate IX and X shows the shape and dimensions of slant shear test specimens.

At first instance, a slant shear specimen is fabricated by constructing a polystyrene of similar slant shape with desired angle (30°) to vertical. In this way, each 200 mm x 100 mm cylinder mould was filled with the fabricated slant shape polystyrene. The remaining half slant shape cylinder mould space is then filled with CEM 1 cement concrete (substrate) also called Portland cement concrete (PCC) to form a PCC substrate slant shape specimen as presented in Plate X. Thus, the substrate specimen cast was demoulded after 24 hours; a slant shape cylinder specimen made of concrete was obtained.

Three types of diagonal bond interface on the substrate layer with the desired angle (30°) to vertical for the slant shear test, which are (a) the smooth surface, (b) the grooved surface and (c) Mesh surface where made on the different cast substrate slant

cylindrical specimen as required after 24 hours of demoulding. Then the cast and surface treated substrate specimen are water cure for 28 days. After 28 days of curing the substrates in the proper environment (water curing), the cast substrate slant cylindrical specimen are filled back into the 200 mm x 100 mm cylindrical mould to fabricated a full cylindrical specimen. The half slant shaped space left in the cylinder is then filled with the geopolymer mortars to form a full cylinder for the slant shear test.

In this study, there are three mix designs, and it is necessary to test the slant shear specimens for 28 and 56 days of age. Thus, a total of 72 substrates are needed. It is necessary to mention that each substrate layer must be at the age of 28 days while casting each mortar mix (overlay). After testing the slant shear specimens, the compression machine shows a number as the applied force or the compressive load applied on the specimen. Thus, the Mohr-Coulomb relations are used to derive the shear and normal stress applied to the specimen. Plate XI presents the Mohr-Coulomb circle adjusted to a slant shear specimen.

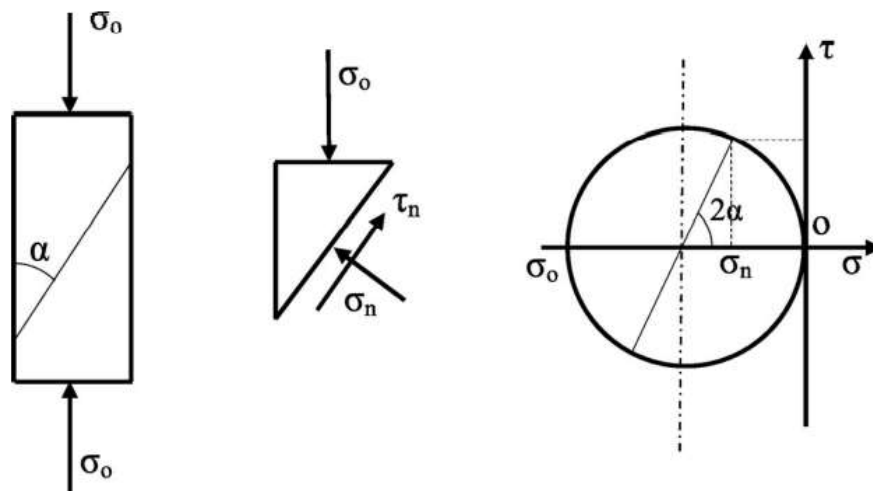


Plate XI: Mohr-coulomb circle adjusted to a slant shear specimen.

Equations 3.3-3.5, shows how to derive shear stress on the bond surface. In these equations, A is the cross-sectional area of the cylinder, F is vertical failure force, σ_n and τ_n are the normal and shear stress applied to the interface.

$$\sigma_o = F/A \quad (3.3)$$

$$\sigma_n = \sigma_o \sin^2(\alpha) \quad (3.4)$$

$$\tau_n = 0.5 \sigma_o \sin^2(\alpha) \quad (3.5)$$

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Physical Properties of Constituent Materials (CPA, MK & Fine Aggregate)

As shown in Table 4.1, the result indicates the physical properties of the CPA and MK. The result was found satisfactorily as it meets up with the necessary requirement for the production of mortar. The MK (503.9 Kg/m^3) was observed to be lighter than CPA (649.7 Kg/m^3).

Table 4.1: Physical properties of the constituent materials

Physical properties	Test Value (Kg/m^3)	
	CPA	MK
Density	649.7	503.9
Specific gravity	2.3	2.2

4.1.1 Physical properties of fine aggregate

Table 4.2 shows the particle size distribution of the available natural river sand used for the experiment. The fine aggregate conforms to the ASTM C33/C33M (2013) standard grading requirement for fine aggregate. The bulk density of the fine aggregate was determined to be 1699 kg/m^3 . The density value is within $1600\text{-}1700 \text{ kg/m}^3$ maximum allowable value and is in accordance with work Kosmatka *et al.* (2002).

Table 4.2: Physical Properties of Fine Aggregate

Properties	Test value	Max. allowable value (%)	Relevance standard/reference
Material finer than $75\mu\text{m}$	0.4%	3%	ASTM C33/C33M (2013)/ ASTM C117/C117M (2017)
Oven-dry bases, bulk density	1699 kg/m^3	$1600\text{-}1700 \text{ kg/m}^3$	Kosmatka <i>et al.</i> (2002)
Specific gravity	2.59	2.4-3.0	Neville (2011)
Moisture content	0.2	0.05-0.8%	Neville (2011)

4.2 Chemical Composition of Constituent Materials

Table 4.3 present the chemical analysis of oxide percentage quantity of the Pozzolanic constituent materials (PC, MK and CPA).

Table 4.3 Chemical Composition of Constituent Materials

Properties	Constituent Materials			Limits
	PC	MK	CPA	
SiO ₂	19.57	72.39	80.83	} SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃ ≥70
Al ₂ O ₃	5.47	20.35	0.77	
Fe ₂ O ₃	3.24	1.12	1.55	
CaO	64.19	0.01	4.24	
MgO	2.01	0.12	0.05	
LOI	2.98	2.35	0.20	10% max.
K ₂ O	0.45	3.12	5.50	
Na ₂ O	0.26	0.34	0.06	
SO ₃	2.74	-	0.83	4.0% max.
TiO ₂	-	0.90	-	
Mn ₂ O	1.25	0.02	0.05	

The result shows the chemical composition of the constituent materials, the sum of SiO₂, Al₂O₃, and Fe₂O₃ was found to be greater than 70%. This implies that cementitious constituent materials satisfy the chemical requirement for N-class of pozzolana in accordance with ASTM C618 (2018).

4.2.2 Grading of fine aggregate

The particle size distribution shows that the fine aggregate contains a uniformity coefficient (Cu) of 2.6 and coefficient of curvature (Cc) of 1.1 with a fine modulus of 3.6, thus indicating fine sand. Figure 4.1 shows the result of the grading of fine aggregate. The fine aggregate fall between the upper and the lower limit, specified by the ASTM C33/C33M (2013) standard. Hence, the fine aggregate was considered to be well graded.

The curve presented in Figure 4.1 depicts the relationship between the percentages of the fine aggregate passing through each sieve and the sizes of the sieves. The coefficient of uniformity, coefficient of curvature, and fineness modulus for the fine aggregate was calculated from the curve presented in Figure 4.1.

The value of D₆₀, D₃₀, and D₁₀ gotten from the graph were calculated as follow:

$$D_{60} = 0.6, D_{30} = 0.38, \text{ and } D_{10} = 0.23$$

$$\text{Uniformity coefficient (Cu)} = \frac{D_{60}}{D_{10}} = 2.6$$

$$\text{Coefficient of curvature (Cc)} = \frac{(D_{30})^2}{D_{10}D_{60}} = 1.1$$

D₆₀ is the particle size diameter passing 60%. D₃₀ is the particle size diameter passing 30% and D₁₀ is the particle size diameter passing 10%.

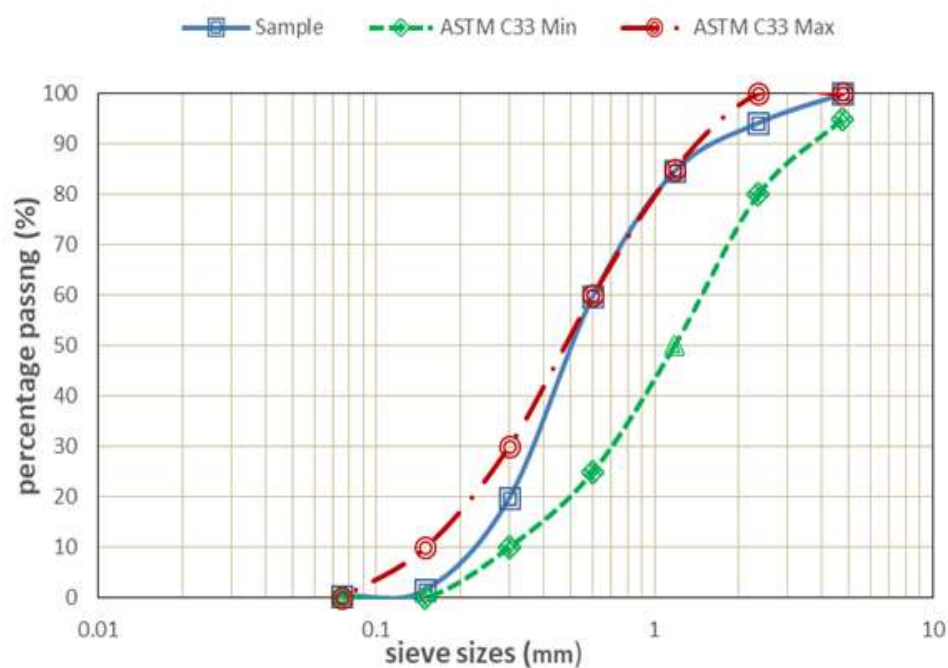


Figure 4.1: Grading of the fine aggregate conforming to ASTM C33/C33M (2013) limit.

4.3. Setting time of the binding materials

4.3.1 Initial and final setting time

In comparing the initial and the final setting time of the mortars, the vicat test was conducted to determine initial and the final setting time. The laboratory results are illustrated in Figure 4.2. The setting time of the geopolymer mortar tested is higher than the cement-based mortar except for the 100% CPA binder material. Figure 4.2 illustrates a graph that demonstrates the initial and final setting time for all mix designs, including cement based and geopolymer mortar. The presence of MK in the geopolymer mix is observed to slow the setting time of the geopolymer mortar in the 50% CPA/ 50% Mk, 100% Mk compared to the 100% CPA which have a shorter setting time. Besides, short setting time is considered as one of the essential properties of the repair mortars. Thus, regardless of other mortar characteristics, the CPA based binders could be an appropriate substitution for OPC binders for using mortars in repairing concrete structures, regarding their shorter setting time. Appendix presents the result of the standard consistency of the mixtures, which ranges from 33-35 mm of the plunger penetration.

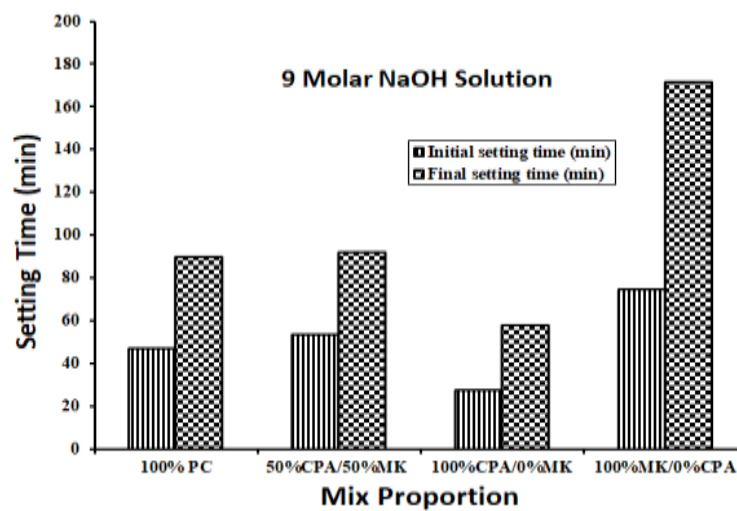


Figure 4.2: Comparison of the setting time in all the mix proportion

4.3.2 Soundness Test of Geopolymer Mortar

The soundness test results were presented in Figure 4.3. Portland cement paste “which was used as a control (100% PC)” showed a lesser expansion as against the binary paste geopolymer mix. Irrespective of the three varying proportions of geopolymer mix, mortars with 100% CPA/0%MK content had higher expansion rate of 3.5 mm compared to others. The mix ratio of 50%CPA/50%MK and 100%MK/CPA0% are having an expansion of 2.5 mm. The expansion of cement was recorded to be 1.00 mm, while those of the geopolymer mix is 2.5 mm and 3.5 mm which is less than the maximum value of 10 mm as presented in BS EN 196-3 (2016).

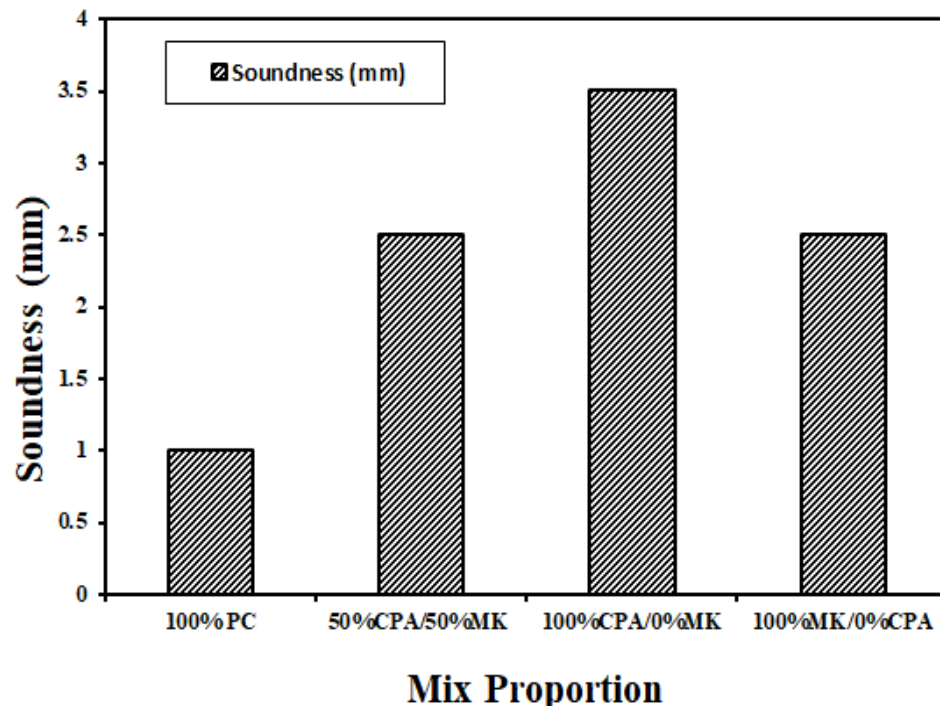


Figure 4.3: Comparison of the Soundness in all the mix proportion

4.3.3 Flowability of Geopolymer Mortar

Flowability of the readily mixed fresh geopolymer mortar was studied by using mini slump test of mortar. Mini slump test (Free flow test) is used to observe the spreading of fresh geopolymer mortar by free flow of mortar on a leveled surface. Throughout the experiment, the rapid setting of CPA based geopolymer mortar was noticed. The fresh mortar began to set within about two minute right after thoroughly mixing process. The width of mortar spreading is shown in Table 4.4

Table 4.4: Mini slump test (free flow test) of geopolymer mortar

MIX ID	Spread Width (mm)
100% PC	123
50% CPA/50% MK	150
100% CPA/0% MK	160
100% MK/0% CPA	163

Increasing trend in the spreading widths is observed with the introduction and increase in replacement level of the precursor material (CPA/MK) in the mortar mixture. 100 CPA/0%MK and 50%CPA/50%MK exhibits faster initial and final setting times of geopolymer mortar. Hence, CPA contributed in reducing the workability and accelerate the geopolymerization of specimen (Gao, Yu and Brouwers, 2016). Lower flowability achieved by CPA as compared to the higher flowability of MK is attributed to the higher content of CaO (4.24) exhibited by CPA and lower content of CaO (0.01) exhibited by MK which thus influence and accelerate the geopolymerization as it leads to its rapid reaction with alkaline activator (Khan *et al.*, 2016).

4.4 Results from the Experiments on the Hardened Mortar

This section aims to elaborate on the compressive and slant bond strength of the mortar while comparing them in mix designs to represent the differences between CEM 1 (control) and CPA/MK based geopolymer mortars. Compressive and Bond strength of mortar are considered as one of the conclusive strength properties that can act as the primary role in the functionality of mortar in practical use.

4.4.1 Compressive strength of Geopolymer Mortar

Table 4.5 presents the compressive strengths of CPA and MK blended geopolymer mortar. The results show that the cube compressive strength of the geopolymer mortar decreased as 100% CPA was introduced to the mix, and likewise as 100% MK was used in the mix.

Table 4.5: Compressive strength for cubic specimens (N/mm²)

MIX ID	Molar	3 days	7 days	28 days	56 days
100% PC		20.50	25.70	37.40	41.60
50%CPA/50%MK		12.00	15.50	18.05	21.78
	9M				
100%MK/0%CPA		5.10	5.70	7.42	7.82
100%CPA/0%MK		7.40	8.65	11.03	14.46

Figure 4.4 illustrates the variation of compressive strength of the geopolymer mortar mixtures containing 50% CPA/50% MK, 100% MK and 100% CPA with respect to that of the control mix (CEM 1 mortar) also referred to as CEM 1 Portland Cement (PC). Comparing the value of the control mix (100% PC), i.e., mortar without any geopolymer

content, the geopolymer mix 50% CPA/50% MK, 100% CPA and 100% MK decreased the compressive strength by 51.7%, 70.5% and 80.2%, respectively at the age of 28 days. Further reductions in compressive strength of 41.5%, 39.7%, 51.7% and 47.6% compared to the PC mortar were observed in mortar containing 50% CPA/50% MK for the curing times of 3, 7, 28 and 56 days, respectively. In geopolymer mortar mixtures containing 100% CPA and 0% MK, the compressive strength values decreased by 63.9%, 66.3%, 70.5% and 65.2% compared to that of PC mortar for the curing times of 3, 7, 28 and 56 days, respectively. The reduction in compressive strength of the mixes containing MK and CPA is attributed to the delay in hydration and slow pozzolanic activity of the geopolymer solid, which negate the increase in compressive strength.

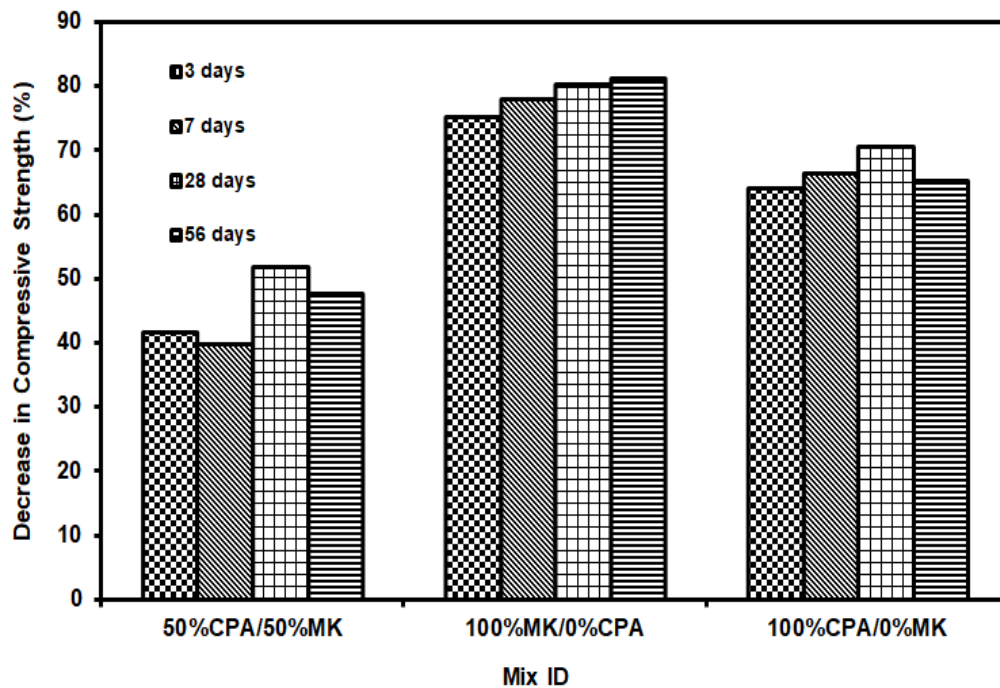


Figure 4.4: Variation in compressive strength of geopolymer mortar with respect to control mix.

Table 4.5 further shows that compressive strength in the geopolymer based mix design (50% CPA/50% MK, 100% MK and 100% CPA) is lower than the cement-based mortar

(100% PC). Although, the compressive strength in the geopolymer mortars shows a sharp growth by introducing equal amount of MK and CPA (50% CPA/50% MK), but when using only MK or CPA (100% CPA or 100% MK), the results shows a drop in compressive strength. The incremental development observed in the compressive strength over the varying hydration period could be a result of the pozzolanic effect (Varma *et al.*, 2017).

Geopolymer chemical reactions of CPA and MK with silicate ions (which is provided by desolving the CPA and MK in the activator) leads to producing C-(A)-S-H gel with a low ratio of Ca/Si. The pozzolanic effect of CPA and MK increases the C-S-H gel in the binder, which strengthens the aggregate's interaction with binder (Varma *et al.*, 2017).

4.4.2 Bond Strength

In this research, the bond strength of mortar is examined using the slant shear test, in which the procedure is explained in sections 3.3.1.3

4.4.2.1 Influence of geopolymer precursor on the interfacial bond strength of the composite

Table 4.6-4.8 illustrates the test results and the calculated shear capacity of the specimens along with the bond interface (τ_n), thus demonstrating the bond strength of the specimens.

To simplify the comparison between the collected experimental data, the bar chart demonstrates that the cement-based (100% PC) mortar shows a better cohesiveness to the substrate layer of concrete than the 100% CPA and 100% MK mortar. However, the bond strength of 50%CPA/50%MK (equal percentage) geopolymer mortars was observed to be higher than the cement based mortar. This growth of bond strength in blended CPA/MK

Geopolymer mortar can be as a result of the increment of C-S-H gel by reason of increasing the percentage of silica (see Table 4.1).

Table 4.6: Slant shear test results of groove interface specimens

Groove Mix ID	28 Days			56 Days		
	σ_0 (N/mm ²)	σ_n (N/mm ²)	τ_n (N/mm ²)	σ_0 (N/mm ²)	σ_n (N/mm ²)	τ_n (N/mm ²)
100% PC	9.42	9.23	4.08	13.76	13.49	5.96
50%CPA/50%MK	13.81	13.53	5.98	16.86	16.53	7.31
100% MK /0% CPA	6.02	5.90	2.61	8.74	8.57	3.79
100% CPA /0% MK	7.02	6.88	3.04	11.13	10.91	4.82

Table 4.7: Slant shear test results of mesh interface specimens

Mesh Mix ID	28 Days			56 Days		
	σ_0 (N/mm ²)	σ_n (N/mm ²)	τ_n (N/mm ²)	σ_0 (N/mm ²)	σ_n (N/mm ²)	τ_n (N/mm ²)
100% PC	8.01	7.8498	3.46833	10.67	10.4566	4.62011
50%CPA/50%MK	9.64	9.4472	4.17412	12.05	11.809	5.21765
100% MK /0% CPA	4.03	3.9494	1.74499	6.84	6.7032	2.96172
100% CPA /0% MK	4.96	4.8608	2.14768	7.46	7.3108	3.23018

Table 4.8: Slant shear test results of smooth interface specimens

Smooth Mix ID	28 days			56 days		
	σ_0 (N/mm ²)	σ_n (N/mm ²)	τ_n (N/mm ²)	σ_0 (N/mm ²)	σ_n (N/mm ²)	τ_n (N/mm ²)
100% PC	5.57	5.46	2.41	7.92	7.76	3.43
50%CPA/50%MK	7.22	7.08	3.13	10.28	10.07	4.45
100% MK/0%CPA	2.39	2.34	1.04	5.92	5.80	2.56
100% CPA/0%MK	3.46	3.39	1.50	6.03	5.91	2.62

Geopolymer is formed using precursors containing alumina (Al₂O₃) and silica (SiO₂) such as CPA and MK activated with alkali solutions. The Al₂O₃ and SiO₂ in the precursor thus dissolve and form a three-dimensional amorphous aluminosilicate network (Davidovits,

1994). Nevertheless, the silicate network of geopolymer which composed of tetrahedral anions $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$ need positive ions such as Na^+ , K^+ , Li^+ , Ca^{++} , Na^+ , Ba^{++} , NH_4^+ , H_3O^+ to compensate the electric charge of Al^{3+} in tetrahedral coordination as shown in Figure 4.5. When geopolymer is patch against the spalled of the reinforced concrete structure made from Ordinary Portland Cement (OPC), geopolymer is chemically bond to substrate due to the reaction products at the interface transition zone between OPC substrate (Somna *et al.*, 2012; Temuujin *et al.*, 2009).

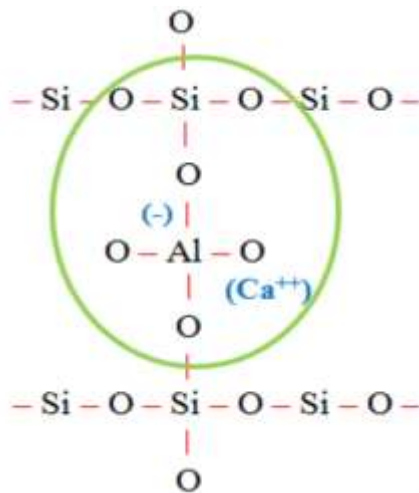


Figure 4.5: Schematic representation of the chemical bond at interface.

Calcium hydroxide or portlandite, which is available at the Ordinary Portland Cement (OPC) substrate surface, provides positive ion Ca^{++} and this positive ion is required for the geopolymerization process. Theoretically, the Ca^{++} was balance the negative charge of Al_3^+ ions in geopolymer thus, promoting the bond strength between geopolymer repair material and substrate. It is necessary to mention that the shear stress applied to the interface is a combination of tension and compressive stresses. Therefore, in a tension-weak material like concrete or mortar, the failure is usually occurring because of tensile cracking rather than

compression and shear slipping. Thus, it is obvious that the pattern of growing bond strength varies from what it was in compressive strength.

4.4.2.2 Influence of substrate surface treatment on the interfacial bond strength of CPA/MK geopolymer mortar

Substrate surface treatment or surface roughness is an important parameter to put into consideration in order to enhance the bond strength between repair material (CPA/MK geopolymer) and concrete substrate as shown in Plate IX and X such as Grooved surface texture, Smooth (As Cast) surface texture and Mesh (Wire Brush) surface texture. The surface of the concrete structure or damaged concrete surface area is treated by increasing the roughness surface and it is believed that it was enhance the bond strength between an old concrete and repair material (Momayez *et al.*, 2005

The interfacial shear bond strength results for specimens using three types of surface texture are illustrated in Figure 4.6 to 4.8.

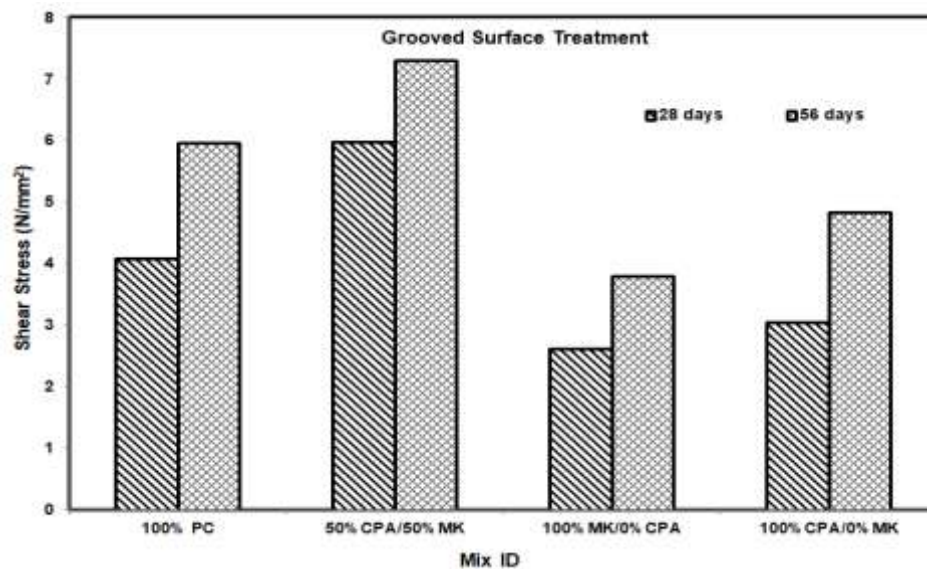


Figure 4.6: Comparison of the bond strength of grooved substrate surface

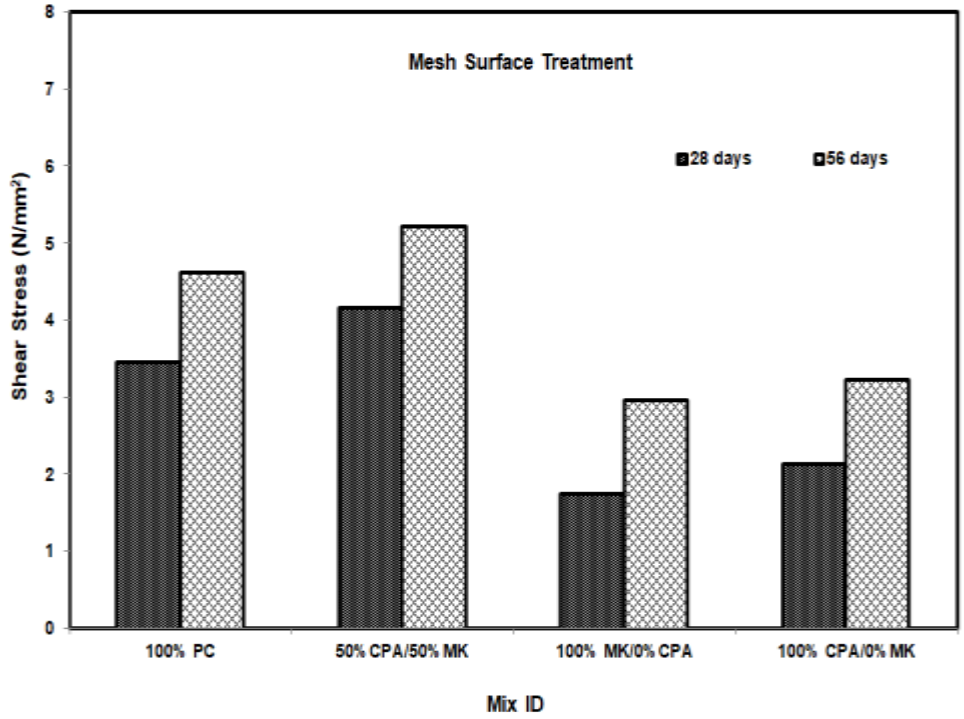


Figure 4.7: Comparison of the bond strength of mesh (wire brush) substrate surface

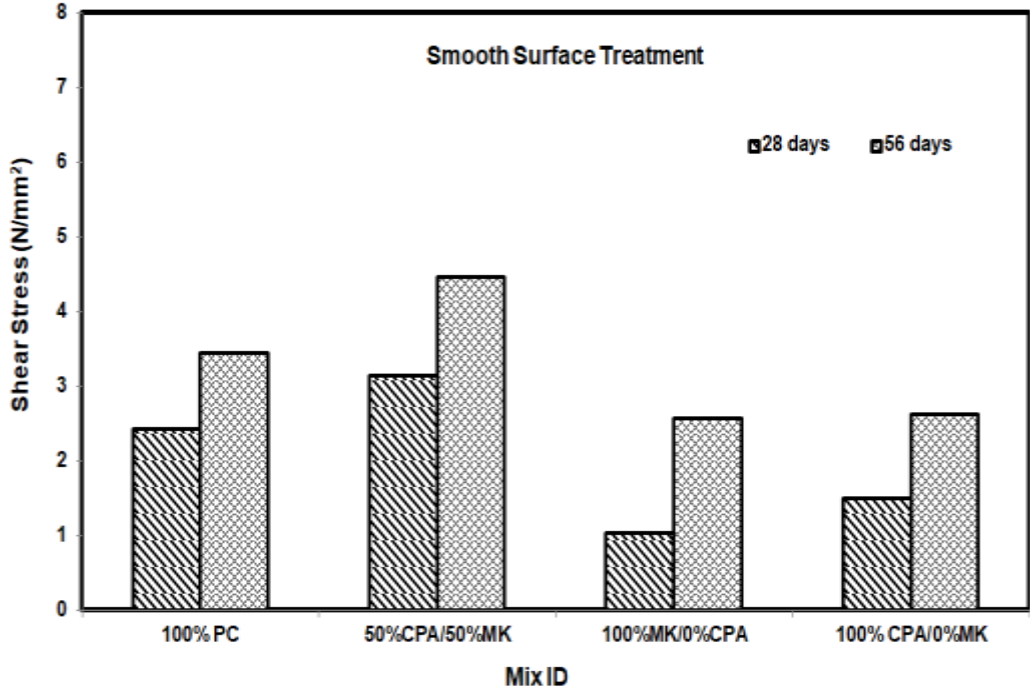


Figure 4.8: Comparison of the bond strength of smooth (As cast) substrate surface

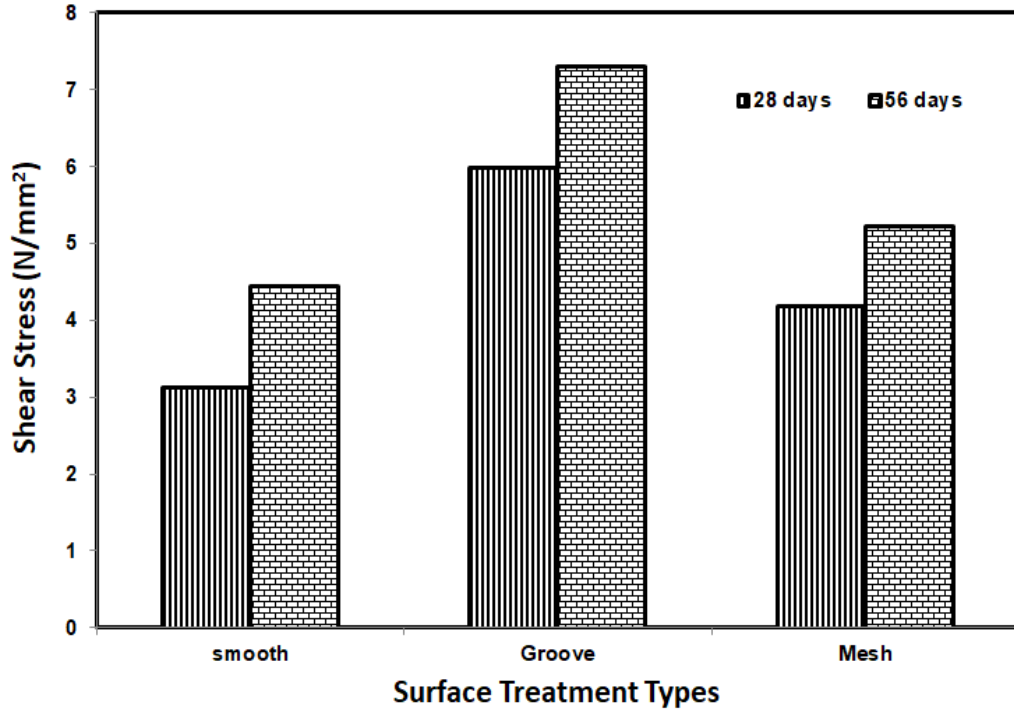


Figure 4.9: Influence of substrate surface treatment on the interfacial bond strength of CPA/MK geopolymer mortar

The experimental results showed that the interfacial bonding for almost all the grooved surface specimens examined in this research was generally good and strong enough as the interfacial failure mostly occurred after the substrate experiencing some degree of damages. In some of the specimens, the bond strength was distinctively stronger than that of the cement based mortar because failure occurred mainly in the substrate without interfacial separation or debonding between the concrete substrate and the geopolymer. The observations in Figure 4.9 indicate significant difference of about 35% between the results for Grooved, Mesh (Wire Brush) and Smooth (As Cast) surface texture.

Figure 4.9 presents the slant shear test results corresponding to different surface texture at different geopolymer mix of the specimens. The smooth surface (As Cast specimens) texture, the average measured shear strengths were 3.42, 4.45, 2.61 and 2.56 N/mm² at 56

days curing for 100% PC, 50%CPA/50%MK, 100%CPA/0%MK, 100%MK/0%CPA geopolymer mortar, respectively. The grooved substrate surface specimens with 50%CPA/50%MK overlay recorded the highest 56 day average shear bond strength of 7.30 N/mm² followed by specimens with mesh (wire brushed) substrate surface and 50%CPA/50%MK overlay, with shear bond strength of 5.2 N/mm², respectively. Hence, the results clearly portray that the different surface preparation methods undertaken, significantly improved the shear bond strength of the composite samples when compared with the As Cast reference samples. The tremendous enhancement in the shear bond strength could generally be attributed to greater adhesion and interlocking between the geopolymer mortar overlay and the roughened normal concrete substrate surfaces.

4.4.2.3 Interfacial transition zone through Scanning Electron Microstructure (SEM)

The interfacial transition zone through Scanning Electron Microstructure (SEM) shows that geopolymer made from CPA and MK is chemically bonded to the concrete substrate as shown in Plate XI. Moreover, the increase in Ca⁺⁺ ion balanced the negative charge of Al³⁺ ions, which leading to a dense interface zone as shown in Figure 10. Therefore, the microstructure images give clear picture in order to evaluate the bond strengths of geopolymer repair materials. Geopolymer made with CPA and MK is good candidates as an alternative bonding material for repair works.

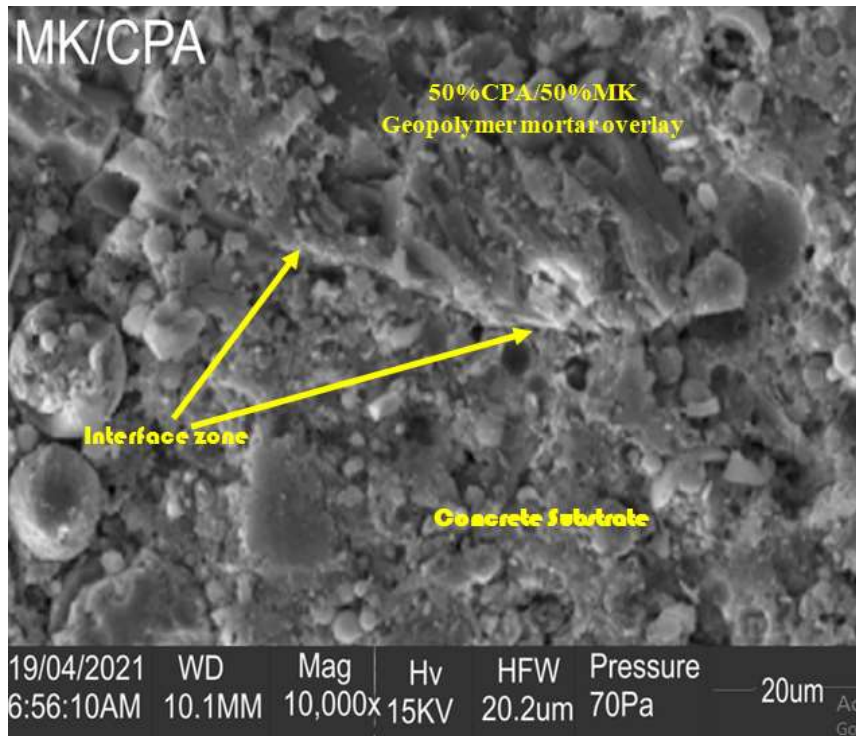


Plate XI: SEM micrographs of interfacial transition zone between geopolymer and concrete substrate.

The microstructures image of these geopolymer, concrete substrate and interface could be identified from unreacted CPA and MK, agglomeration of Portland cement and small line crossing along the interfacial, respectively. The negatively charged Al and tetrahedrally coordinated Al (III) atoms inside the network are charge-balanced by alkali metal cations such as Na^+ or K^+ (from alkaline activator) and Ca^{++} (from Portland cement grains reacting with water). Thus, geopolymer which is rich in Si^{4+} and Al^{3+} ions can react with $\text{Ca}(\text{OH})_2$ at the surface of substrate leading to bonding strength development at the contact zone.

4.5 Summary of Findings

1. The CPA/MK based mortar is noticeably more flow-able/workable in comparison with equivalent cement-based mortar.
2. The initial and final setting time in 50%CPA/50%MK, 100%CPA/0%MK geopolymer repair mortar is considerably shorter than cement-based mortar, which can be in most cases beneficial for using this type of mortar in repairing concrete structures.
3. 100%CPA/0%MK geopolymer mix delays the initial setting time to 25 minutes, and the final setting time to 60 minutes.
4. In this research, substituting 50%CPA and 50%MK by weight of binder (precursor) in geopolymer mortar is the optimum amount to achieve the good compressive strength and enhanced slant shear bond strength.
5. Grooved surface treatment method gave the highest slant shear bond strength with 7.31 N/mm² value.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This experiment's primary goal is to produce an alkali-activated mortar without using cement and with appropriate characteristics for repairing concrete structures. The functionality of this mortar in repairing the concrete structures can extend the lifetime of existing structures. Since the proper workability and setting time are considered two essential characteristics of repair mortar, the effect of precursor materials (CPA and MK) were evaluated on the geopolymer mix by carrying out flowability and vicat test. After that, compressive strength of mortar at the ages of 3, 7, 28 and 56 days with $100 \times 100 \times 100$ mm cubic specimens is evaluated. Moreover, the bond strength of mortar was evaluated using the slant shear test with 100×200 mm cylindrical specimens at 28 and 56 days. The compressive strength and slant shear properties of the interface between plain concrete substrate and CPA/MK geopolymer mortar showed that the bond strength in the slant shear test was very strong, as the interfacial failure occurred in the plain concrete substrate without interfacial separation or debonding between the plain concrete substrate and geopolymer mortar. The CPA/MK based mortar is noticeably more flow-able/workable in comparison with equivalent cement-based mortar. The initial and final setting time in 50%CPA/50%MK, 100%CPA/0%MK geopolymer repair mortar is considerably shorter than cement-based mortar, which can be in most cases beneficial for using this type of mortar in repairing concrete structures. 100%MK/0%CPA geopolymer mix delays the initial setting time to 25 minutes, and the final setting time to 60 minutes. 50%CPA and 50%MK by weight of binder (precursor) in geopolymer mortar is the optimum amount

regarding compressive strength. Grooved surface treatment method gave the highest slant shear bond strength with 7.31 N/mm² value.

5.2 Recommendations

The following are recommended from the outcome of this experiment.

1. Towards tackling waste management issues and forth with reduce CO₂ effect on the environment, geopolymer precursor from agricultural waste and earth explore source (CPA and MK) are recommended in geopolymer mortar mix meant for concrete repair.
2. In areas of large cassava farming and high deposits of naturally occurring clay mineral whose main component of kaolin is the mineral kaolinite. The use of CPA and MK is recommended for the production of binder for construction works.

5.3 Contribution to Knowledge

Considering all inferences and appraisals from this research, 50%CPA and 50%MK blended precursors in geopolymer mortar with grooved surface treatment type are recommended in the production of retrofitting/repair concretes with the sole aim of achieving enhanced interfacial shear bond strength.

5.4 Area of Further Study

1. Further research should look into the use of different grooving method, such as zig-zag grooving, double directional grooving etc.
2. Also, further research work should be conducted to provide a simplified formula to evaluate the shear bond strength in terms of the relative compressive strength of concrete and the relative shear bond strength for the different bonding surface conditions.
3. The possibility of using alkali-activated mortars in 3D printers should be evaluated.

4. Using Nano powders and assessing the effectiveness of using them on the physical and mechanical properties of CPA/MK-based mortars should be examined.
5. Further research should also look into the substitution of MK and CPA at other varying ratios.

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APPENDICES

APPENDIX A

Table A1: Fresh Properties of Mortar

Mix ID	Consistency (mm)	Initial setting time (min)	Final setting time (min)	Soundness (mm)
100% PC	33	47	90	1
50% CPA/50% MK	35	54	92	2.5
100% CPA/0% MK	33	28	58	3,5
100% MK/0% CPA	34	75	172	2.5

Table A2: Direct, Normal, and Shear stress applied to the interface on Smooth surface of 28 days Curing

Mix ID	Direct stress applied (σ_o)	Normal stress applied to the interface. (σ_n)	Shear stress applied to the interface. (τ_n)
100% PC	5.57	5.4586	2.41181
50% CPA/50% MK	7.22	7.0756	3.12626
100% MK/0% CPA	2.39	2.3422	1.03487
100% CPA/0% MK	3.46	3.3908	1.49818

Table A2: Direct, Normal, and Shear stress applied to the interface on Smooth surface of 56 days Curing

Mix ID	Direct stress applied (σ_o)	Normal stress applied to the interface. (σ_n)	Shear stress applied to the interface. (τ_n)
100% PC	7.92	7.7616	3.42936
50% CPA/50% MK	10.28	10.0744	4.45124
100% MK/0% CPA	5.92	5.8016	2.56336
100% CPA/0% MK	6.03	5.9094	2.61099

Table A3: Direct, Normal, and Shear stress applied to the interface on Groove surface of 28 days curing

Mix ID	Direct stress applied (σ_o)	Normal stress applied to the interface. (σ_n)	Shear stress applied to the interface. (τ_n)
100% PC	9.42	9.2316	4.07886
50% CPA/50% MK	13.81	13.5338	5.97973
100% MK/0% CPA	6.02	5.8996	2.60666
100% CPA/0% MK	7.02	6.8796	3.03966

Table A4: Direct, Normal, and Shear stress applied to the interface on Groove surface of 56 days curing

Mix ID	Direct stress applied (σ_o)	Normal stress applied to the interface. (σ_n)	Shear stress applied to the interface. (τ_n)
100% PC	13.76	13.4848	5.95808
50% CPA/50% MK	16.86	16.5228	7.30038
100% MK/0% CPA	8.74	8.5652	3.78442
100% CPA/0% MK	11.13	10.9074	4.81929

Table A5: Direct, Normal, and Shear stress applied to the interface on Mesh surface of 28 days curing

Mix ID	Direct stress applied (σ_o)	Normal stress applied to the interface. (σ_n)	Shear stress applied to the interface. (τ_n)
100% PC	8.01	7.8498	3.46833
50% CPA/50% MK	9.64	9.4472	4.17412
100% MK/0% CPA	4.03	3.9494	1.74499
100% CPA/0% MK	4.96	4.8608	2.14768

Table A6: Direct, Normal, and Shear stress applied to the interface on Mesh surface of 56 days curing

Mix ID	Direct stress applied (σ_o)	Normal stress applied to the interface. (σ_n)	Shear stress applied to the interface. (τ_n)
100% PC	10.67	10.4566	4.62011
50% CPA/50% MK	12.05	11.809	5.21765
100% MK/0% CPA	6.84	6.7032	2.96172
100% CPA/0% MK	7.46	7.3108	3.23018

Table A6: Influence of substrate surface treatment on the interfacial bond strength of CPA/MK geopolymer mortar

Surface type/days	28 days			56 days		
Smooth	7.22	7.0756	3.12626	10.28	10.0744	4.45124
Groove	13.81	13.5338	5.97973	16.86	16.5228	7.30038
Mesh	9.64	9.4472	4.17412	12.05	11.809	5.21765

Table A7: Experimental readings on Slant Shear Bond Test of Smooth surface of Specimen

Mix ID	28 days Curing			56 days Curing		
	Direct stress applied (σ_o)	Cross-sectional area (A)	Vertical failure force (F)	Direct stress applied (σ_o)	Cross-sectional area (A)	Vertical failure force (F)
100% PC	5.57	78539.82	437466.80	7.92	78539.82	622035.37
50%CPA/50%MK	7.22	78539.82	567057.50	10.28	78539.82	807389.35
100% MK/0%CPA	2.39	78539.82	187710.17	5.92	78539.82	464955.73
100% CPA/0%MK	3.46	78539.82	271747.78	6.03	78539.82	473595.11

Table A8: Experimental readings on Slant Shear Bond Test of Groove surface of Specimen

Mix ID	28 days Curing			56 days Curing		
	Direct stress applied (σ_o)	Cross-sectional area (A)	Vertical failure force (F)	Direct stress applied (σ_o)	Cross-sectional area (A)	Vertical failure force (F)
100% PC	9.42	78539.82	739845.10	13.76	78539.82	1080707.92
50%CPA/50%MK	13.81	78539.82	1084634.91	16.86	78539.82	1324181.37
100% MK/0%CPA	6.02	78539.82	472809.72	8.74	78539.82	686438.03
100% CPA/0%MK	7.02	78539.82	551349.54	11.13	78539.82	874148.20

Table A9: Experimental readings on Slant Shear Bond Test of Mesh surface of Specimen

Mix ID	28 days Curing			56 days Curing		
	Direct stress applied (σ_o)	Cross-sectional area (A)	Vertical failure force (F)	Direct stress applied (σ_o)	Cross-sectional area (A)	Vertical failure force (F)
100% PC	8.01	78539.82	629103.96	10.67	78539.82	838019.88
50% CPA/50% MK	9.64	78539.82	757123.86	12.05	78539.82	946404.83
100% MK/0% CPA	4.03	78539.82	316515.47	6.84	78539.82	537212.37
100% CPA/0% MK	4.96	78539.82	389557.51	7.46	78539.82	585907.06

Table A10: Compressive strength for cubic specimens of Portland Cement (PC) and Geopolymer Mortar (GPM) (N/mm²)

MIX ID	Molar	3 days	7 days	28 days	56 days
100% PC		20.5	25.7	37.4	41.6
50% CPA/50% MK	9M	12	15.5	18.05	21.78
100% CPA/0% MK	9M	5.1	5.7	7.42	7.82
100% MK/0% CPA	9M	7.4	8.65	11.03	14.46

Table A11: Variation (decrease) in compressive strength of Geopolymer Mortar with respect to control mix (%).

MIX ID	3 days	7 days	28 days	56 days
50% CPA/50% MK	41.46341	39.68872	51.73797	47.64423
100% MK/0% CPA	75.12195	77.82101	80.16043	81.20192
100% CPA/0% MK	63.90244	66.34241	70.50802	65.24038

APPENDIX B: Plates showing the laboratory procedures



Plate B1: Vicat apparatus for Setting time test



Plate B2: Geopolymer mortar mix



Plate B3: Geopolymer mortar flowability test.
(spread diameter check)



Plate B4: Slant Shear Specimen



Plate B5: Geopolymer Mortar sample oven curing



Plate B6: Geopolymer Mortar cube specimen weighing



Plate B7: Soundness test sample



Plate B8: Plain/smooth surface sample



Plate B9: Groove surface sample



Plate B10: Shear bond test under compression



Plate B11: Cylinder splitting Shear bond test sample



Plate B12: Failure mode of Cylinder splitting Shear bond test sample

