DEVELOPMENT OF POFA BASED GEOPOLYMER CONCRETE ACTIVATED WITH WOOD ASH LYE INCORPORATING TIMBER CLINKER AGGREGATE

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by

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LIST OF SYMBOLS

| А | Cross sectional area |
|------------------|---|
| a | Distance between beam supports |
| b | Width of slab |
| d | Effective depth of slab |
| e | Porosity |
| E | Modulus of elasticity |
| f_c | Compressive strength |
| f _{ck} | Characteristics strength of concrete |
| f_{cy} | Characteristic strength of steel |
| f_f | Flexural strength |
| g | Gravity |
| Н | Overall height of slab |
| Hz | Hertz |
| hr | Hour |
| I_p | Intrinsic air permeability |
| Kg | Kilo gram |
| 1 | Liter |
| L/B | Liquid binder ratio |
| М | Bending moment |
| М | Molar concentration |
| m | Mass |
| m, cm, mm, μm | Meter, centimeter, millimeter, micrometer |
| MPa | Mega pascal |
| N, kN | Newton, kilonewton |

| Р | Maximum load |
|-----------------------|-------------------------------------|
| <i>P</i> ₁ | Gas pressure at top |
| <i>P</i> ₂ | Gas pressure at bottom |
| Sec | Second |
| t | Time |
| V | Volume |
| V | Shear force |
| v | Shear stress |
| W | Weight |
| W/C | Water cement ratio |
| Wdry | Dry weight |
| Wsat | Saturated weight |
| Wwet | Saturated weight of sample in water |
| 5 | Damping ratio |
| ρ | Density |
| °C | Degree Celsius |
| θ | Diameter |
| σ | Tensile strength |
| Л | Pi which is equal to 3.14159 |
| %g | Percentage gravity |

LIST OF ABBREVIATIONS

| AAR | Alkali activator ratio |
|---------|--|
| AAS | Alkali activator solution |
| ACI | American Concrete Institute |
| ASTM | American Association for Testing and Materials |
| BS | British Standard |
| BS EN | The British adoption of European Standard |
| C-A-S-H | Calcium Alumino Silicate Hydrate |
| C-S-H | Calcium Silicate Hydrate |
| EDX | Energy Disperse X-ray |
| FA | Fly Ash |
| FTIR | Fourier Transform Infrared Spectroscopy |
| GCS | Geopolymer Composite Slab |
| GGBS | Ground Granulated Blast furnace Slag |
| G-POFA | Ground Palm Oil Fuel Ash |
| IS | Indian Standard |
| K-A-S-H | Potassium Alumino Silicate Hydrate |
| LVDT | Linear Voltage Displacement Transducers |
| LWA | Lightweight Aggregate |
| LWCCS | Lightweight Cement Concrete Slab |
| LWGCS | Lightweight Geopolymer Concrete Slab |
| L/B | Liquid binder ratio |
| NA | Normal Aggregate |
| OPC | Ordinary Portland Cement |
| PFA | Pulverised Fuel Ash |
| PG | POFA Geopolymer |
| POFA | Palm Oil Fuel Ash |
| POS | Palm Oil Shell |
| RCA | Recycle Concrete Aggregate |
| SEM | Scanning Electron Microscopy |
| SSD | Saturated Surface Dry |

| TC | Timber Clinker |
|--------|-----------------------------|
| TCA | Timber Clinker Aggregate |
| TPOFA | Treated Palm Oil Fuel Ash |
| TPS | Transient Plane Source |
| U-POFA | Ultrafine Palm Oil Fuel Ash |
| UPV | Ultrasonic Pulse Velocity |
| WA | Wood Ash |
| XRD | X-ray Diffraction |
| XRF | X-ray Fluorescence |

LIST OF APPENDICES

- APPENDIX A MIX DESIGN OF WA LYE ACTIVATED POFA GEOPOLYMER MORTAR
- APPENDIX B MIX DESIGN OF LIGHTWEIGHT GEOPOLYMER CONCRETE
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PEMBANGUNAN KONKRIT GEOPOLIMER BERASASKAN POFA DIAKTIFKAN DENGAN PENGALKALIAN ABU KAYU MENGANDUNGI AGREGAT KLINKER KAYU

ABSTRAK

Pembuatan simen telah mengakibatkan pelepasan karbon dioksida (CO₂) yang berlebihan di atmosfera, yang menyumbang kepada cabaran kemampanan global. Tambahan lagi dengan jumlah bahan buangan industri yang banyak seperti abu bahan bakar kelapa sawit (POFA), abu kayu (WA), dan agregat klinker kayu (TCA) yang ditimbus di alam sekitar ditambah lagi dengan penggunaan batu agregat semulajadi juga memberi kesan negatif kepada kemampanan global. Sebagai penyelesaian kepada cabaran kemampanan oleh pengeluaran simen, pengurusan bahan buangan dan penggunaan batu agregat semulajadi, teknologi konkrit geopolimer di jadikan sebagai alternatif. Pembangunan konkrit geopolimer telah mendapat periktirafan kebelakangan ini sebagai inovatif dan alternatif bahan binaan. Walaubagaimanapun penggunaan bahan kimia yang agresif seperti sodium hidroksid (NaOH) sebagai pengaktif alkali dan pengawetan oven telah menghadkan penggunaan teknologi geopolimer dilakukan di makmal sahaja. Kajian ini berusaha untuk menjawab persoalan kajian tentang keberkesanan penggunaan pengalkalian WA sebagai pengaktif alkali menggantikan NaOH dan juga penggunaan TCA sebagai agregat kasar dalam konkrit geopolimer pada suhu ambien. Penyiasatan dimulakan dengan mengoptimumkan cecair/pengikat (L/B) dan nisbah pengaktif alkali (AAR) menggunakan pelbagai nisbah L/B dan AAR. Spesimen dengan GGBS sebagai penggantian separa POFA dari 0% hingga 40% dihasilkan bagi menghasilkan kandungan optimum GGBS. TCA sebagai bahan gantian agregat

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semulajadi dioptimumkan dengan menghasilkan konkrit geopolimer POFA:GGBS dengan 0% hingga 100% TCA pada 20% kenaikan. Ujian sifat segar, mekanikal, ketahanan, ciri-ciri termal dan ketahanan terhadap persekitaran yang agresif telah dijalankan. Parameter yang telah dioptimumkan digunakan dalam penghasilan papak konkrit geopolimer ringan. Hasil kajian mendapati nisbah L/B optimum adalah 0.50 dan AAR optimum adalah 3.0. Spesimen dengan 30% GGBS mempamerkan ciri-ciri segar yang lebih baik dengan peningkatan ciri-ciri mekanikal pada 28 hari dan ciriciri ketahanan sehingga 56 hari. Untuk konkrit geopolimer ringan, spesimen mengandungi 60% TCA mencapai ketumpatan sebanyak 1862.82kg/m³ dan kekuatan mampatan 35.93MPa pada 28 hari iaitu memenuhi spesifikasi ASTM C330 (2009). Papak konkrit bertetulang geopolimer ringan menunjukkan kapasiti menanggung beban yang lebih tinggi berbanding dengan papak konkrit bertetulang ringan dan papak komposit geopolimer. Penggunaan POFA, pengalkalian WA dan TCA sebagai pengikat, pengaktif alkali, dan agregat kasar telah menghasilkan konkrit bertetulang geopolimer ringan yang memenuhi spesifikasi dan boleh digunakan sebagai bahan binaan mampan.

DEVELOPMENT OF POFA BASED GEOPOLYMER CONCRETE ACTIVATED WITH WOOD ASH LYE INCORPORATING TIMBER CLINKER AGGREGATE

ABSTRACT

Cement manufacturing has led to excessive carbon dioxide (CO_2) emission into the atmosphere, which has contributed to global sustainability challenges. Moreover, huge amount of industrial waste like palm oil fuel ash (POFA), wood ash (WA), and timber clinker aggregate (TCA) landfilled in our environments coupled with the use of naturally occurring rocks for aggregate are also a negative factor for the global sustainability. Amidst these sustainability challenges of cement production, waste management, and the use of naturally occurring rocks emerges the technology of geopolymer concrete. The development of geopolymer concrete has gained recognition in recent years as innovative and alternative construction material. However, using aggressive chemicals like sodium hydroxide (NaOH) as alkaline activator and oven curing has limited it to laboratory research only. The current study strived to answer the research questions on the effectiveness of using WA lye as alkaline activator to replace NaOH and also the utilization of TCA as coarse aggregate in geopolymer concrete at the ambient temperature. The investigation began with the optimization of liquid binder (L/B) and alkali activator ratio (AAR) using varying L/B and AAR ratio. Then specimens with GGBS as a partial replacement of POFA from 0% to 40% were produced with the view to optimize the GGBS content. TCA was optimized by producing POFA: GGBS geopolymer concrete with 0% to 100% TCA at 20% increment as partial replacement of natural aggregate. The fresh, mechanical, durability, thermal properties, and resistance to

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aggressive environment tests were conducted. The optimized parameters were used to produce lightweight geopolymer slabs. The study revealed that the optimum L/B ratio was 0.50 and the optimum AAR was 3.0. The specimen with 30% GGBS exhibited better fresh properties with an enhance mechanical properties at 28 days and durability properties at up to 56 days. For lightweight geopolymer concrete, specimens with 60% TCA attained the 28 days density and compressive strength of 1862.82kg/m³ and 35.93MPa, respectively, fulfilling the requirements of ASTM C330 (2009). The lightweight reinforced geopolymer concrete slab showed a higher load carrying capacity compared to lightweight reinforced concrete slab and geopolymer composite slab. The use of POFA, WA lye and TCA as binder, alkaline activator, and coarse aggregate produced a lightweight reinforced geopolymer concrete to fulfill the specification and can effectively be utilized as a sustainable construction material.

CHAPTER 1

INTRODUCTION

1.1 Background of study

The Malaysian economy is fast growing which call for the excessive demand of energy in the country. According to Petinrin & Shaaban (2015) energy supply is expected to increase from 65.9% in 2015 to 130.5% Mega ton of oil equivalent (Mtoe) in 2030 at a growing rate of 2.8% per annum, mainly from fossil fuel. To reduce the over dependency of the country's fossil fuel, palm oil and wood industries considered the use of biomass energy as possible alternative (Ahmad & Tahar, 2014). The continues growth of these industries has led to the generation of enormous wastes annually. These wastes materials such as palm oil fuel ash (POFA), wood ash (WA), and timber clinker (TC), cause environmental and health challenges as well as financial losses to the industries. POFA is obtained from burning of end products of oil extraction process from fresh palm fruits, these end products are husk and shell which are burnt in palm oil mill at a temperature of 800 - 1000°C to produce steam used in turbine engine for electricity production (Salam et al., 2018). This ash is dumped in an open field creating environmental and health hazard. It was reported that 10 million tons of POFA is produced each year in Malaysia (Hamada et al., 2018). Similarly, it was reported that wood industries in Malaysia generates large volume of WA and TC annually which account to 45 - 50% of the total volume of the saw log input (Ramasamy et al., 2015). In recent years, immense efforts have been made to accomplish sustainability in construction sector by recycling these and other waste materials in order to reduce excessive cement utilization and also in the production geopolymer concrete (Awang et al., 2016; Awang & Arminda, 2019; Cheah, Part, et al., 2017; Huseien et al., 2016; Jaturapitakkul et al., 2011; Kabir et al., 2015; Ken et al., 2015; LI & LI, 2018; Salih et al., 2015a; Stolz et al., 2019).

Although the technology of geopolymer concrete is gaining recognition globally, it faced some challenges which hinder its wide range application in the construction sector. These challenges include the use of highly concentrated chemical for its activation and the need for high temperature (oven) curing. The high cost of these chemical activators coupled with excessive energy requirement for oven curing of geopolymer concretes confined it to mainly laboratory research and not applicable in actual construction process due to its high cost of production compared to conventional concrete.

To eliminate the use of the oven curing several researches were carried out to include the use of high calcium content binder in the geopolymer matrix such as ground granulated blast furnace slag (GGBS), steel slag powder (SSP), high calcium fly ash (HCFA) and ordinary Portland cement (OPC). According to Suwan & Fan, (2014), a fly ash (FA) based geopolymer concrete cannot set within 24 hours of production at an ambient temperature, which requires the addition of some materials with high content of calcium to accelerate the setting time of the geopolymer cement. They further stated that the required calcium content could be achieved by adding GGBS, HCFA, SSP, or OPC. Also Li et al. (2018) reported that geopolymer containing high calcium binders set in less than 30 minutes whereas those produced with low calcium binders took couple of days to set.

This study attempted to integrate these industrial wastes in the production of WA lye activated POFA lightweight geopolymer concrete cured at ambient temperature. Within four phases, the study was designed to achieve the optimum mixture of lightweight geopolymer concrete that meet the requirement of ASTM C330 (2009). In the first and second phases of the study, WA lye and GGBS content have been optimized to produce an ambiently cured POFA geopolymer mortar with an enhanced fresh, mechanical, and durability properties. The third phase of the study incorporates timber clinker aggregate (TCA) as partial replacement of crushed granite. In this phase, the rheological, mechanical, durability, and thermal properties were examined. The optimum mixture from the previous phases were used to produce and evaluate lightweight geopolymer concrete slab (LWGCS), lightweight cement concrete slab (LWCCS), and geopolymer composite slab (GCS).

1.2 Statement of research problem

Geopolymer concrete has been widely researched as a viable alternative to the existing portland cement concrete (Provis, 2014). Although, the technology of geopolymer is a welcome development but has been faced with some challenges which limited it to laboratory research only and not widely applicable onsite. These challenges include the use of chemical activators and the requirement for oven curing which is energy intensive. This prompts several researchers to investigate the suitability of using agricultural and industrial wastes such as POFA, WA, GGBS etc in the geopolymer concrete production.

POFA obtained from burning of end products of oil extraction process from fresh palm fruits, these end products burnt in palm oil mill are dumped in an open field creating environmental and health hazards. In Malaysia alone, it was reported that 10 million tons of POFA are produced per year (Hamada et al., 2018). The utilization of POFA in the fabrication of geopolymer concrete was investigated and results show the suitability of this waste material as a co-binder (Tudin et al., 2018; Fahim et al., 2016; Islam et al., 2014; Khankhaje et al., 2016; Kubba et al., 2018; Monita et al., 2017; Lim et al., 2018). Unfortunately, most of the studies focused on the activation of POFA geopolymer with NaOH or potassium hydroxide (KOH), this makes it more expensive. Therefore, the possibility of using a strong alkaline (WA lye) solution needs to be investigated to provide an alternative to the high-cost chemical activators used in geopolymer concrete production.

It was reported that from 1992 to 2010, about 98.2 x 10⁷ m³ of wood waste including WA and TC were generated globally (Tamanna et al., 2020). In Malaysia, it was reported that wood industries were among the fastest-growing manufacturing industries and are considered generators of a vast number of wastes (Ramasamy et al., 2015). Part of the way of minimizing the wood wastes is its utilization as biomass energy in the generation of electricity for the consumption of the wood companies or other industries. This process produced more hazardous wastes as in WA, which may cause environmental and health problems if not carefully handled (Mangi, 2017). Today, due to limited studies to incorporate these wastes materials in the construction sector, the major wastes management practice by the generating industries remains landfill disposal causing both environmental and health issues directly affecting global sustainability.

Moreover, the oven curing method of geopolymer concrete has been identified as one of the major challenges of this greener construction material confining it to laboratory research only. Although many researchers concluded that the use of high calcium materials such as OPC, GGBS, and metakaolin in binders with low calcium content results in better performance in terms of setting, hardening, and overall strength when cured at ambient temperature (Al-Majidi et al., 2016; Nath & Sarker, 2014; Salih et al., 2015a; Wattanachai & Suwan, 2017). However, the effect of high calcium content binder such as GGBS in the newly developed geopolymer matrix activated with WA lye need to be investigated.

Another sustainability challenge is natural resource depletion which is a negative factor for the environment. The continued excavation of naturally occurring rocks for coarse aggregates will lead to their depletion in the future (Zawawi et al., 2020). As a result of this many researchers have shifted their focus to the use of waste materials such as palm oil clinker aggregate (POCA), recycled concrete aggregate (RCA), TCA, and palm oil shell (POS) in the manufacturing of concrete. TCA has started gaining recognition as supplementary material for natural aggregate in concrete production (Jung et al., 2017). However, no research was conducted on its usage in the production of geopolymer concrete. Hence, the effect of TCA on the fresh, mechanical, and durability properties of geopolymer concrete need to be investigated.

The use of waste materials in geopolymer concrete production can reduce the negative environmental effects related to concrete production and provide a means for managing these wastes materials which could otherwise be landfilled. This will help in achieving the sustainable development goal (SDG) 12 which aimed to bring a better quality of life by minimizing the use of natural resources and toxic materials as well as the emissions of waste, and also SDG 13 which aimed at reducing climate change and its impacts on our environments. Therefore, this research investigates the potential to form a POFA lightweight geopolymer concrete activated with WA lye incorporating TCA as coarse aggregate under ambient curing conditions. Using WA lye will eliminate the use NaOH in geopolymer concrete production and the incorporation of TCA will also reduce the excavation rate of our virgin rocks for aggregate production thereby contributing to the environmental sustainability.

1.3 Research questions

The following questions were formulated in this study, this is with a view to propose a solution to the current challenges of geopolymer concrete production which confined it to laboratory research only and to increase the recycling of POFA, WA, and TCA wastes generated by palm oil and timber industries.

- 1. What are the suitable mix compositions of WA lye activated POFA geopolymer mortar?
- 2. What will be the effect of GGBS on rheological, mechanical, and durability properties of WA lye activated POFA geopolymer mortar cured at ambient temperature?
- 3. Can TCA be effectively utilized to partially replace crushed granite as coarse aggregate in ambiently cured POFA: GGBS lightweight geopolymer concrete activated with WA lye?
- 4. Can the WA lye activated POFA: GGBS lightweight geopolymer concrete produced with TCA be utilized as a reinforced concrete or composite slab?

1.4 Aim and objectives

The main aim of this research is to investigate the possibility of using WA lye as an alkaline activator alongside with sodium silicate (Na₂SiO₃) in the activation of lightweight POFA geopolymer concrete incorporating timber clinker as partial replacement of coarse aggregate.

The objectives of the research include:

1. To develop a suitable POFA geopolymer mix composition utilising WA lye as alkaline activator.

- To determine the rheological, engineering, and durability performance of the WA lye activated geopolymer mortar using binary mix of POFA and GGBS under ambient curing temperature.
- 3. To investigate the properties of fresh and hardened lightweight geopolymer concrete incorporating TCA as partial replacement of coarse aggregate.
- 4. To determine the strength, loading characteristics, and vibration response of reinforced lightweight geopolymer concrete and composite slabs.

1.5 Significance of the research

The research was carefully designed with the sole aim of providing a sustainable and green construction material with major focused on environmentally friendly and waste management. The introduction of renewable energy sources for electricity generation as a way of reducing the over dependency on fossil fuel to generate electricity in Malaysia has yielded the introduction of new waste materials that need proper handling to reduce its environmental effect. These wastes materials include WA and TC generated from timber industries (Lin et al., 2020), and POFA, a waste generated from palm oil industries which account for almost 10 million tons produced per annum in Malaysia (Hamada et al., 2018).

The successful completion of this research will provide an alternative for the presently used NaOH as alkaline activator in the production of geopolymer concretes, which will not only reduce the production cost because of eliminating the use of the NaOH but will also provide means of utilizing the wastes from wood industries, thereby converting wastes to wealth. The reduction in the cost of production of the geopolymer concrete will also trigger its massive production and will promote the use of the huge amount of wastes generated by Malaysian wood and

palm oil industries as a binder and coarse aggregates in the fabrication of the geopolymer concrete. This will in turn reduce cost of construction projects, reduce the greenhouse gas emission as a result of cement production and provide means for managing wastes generated by the two major industries in Malaysia.

The utilization of alkaline activator generated from WA in the fabrication of POFA geopolymer concrete was not given attention from researchers, because the research on utilizing WA as an alkaline activator in geopolymer concrete is at its infant stage. The data to be derived from this research will be beneficial in increasing the body of knowledge in this area of study and will also reduce the production cost of geopolymer concrete.

The study also intent on utilizing the POFA geopolymer in the production of lightweight geopolymer concrete slab. A new and green construction material to be produced with almost 80% waste materials. The low cost of this newly introduced construction material will boost the mass housing project in countries like Malaysia and Nigeria who are suffering from high cost of construction materials.

1.6 Scope of the study

The study assessed the effectiveness of using the WA lye as an alkaline activator in the production of POFA based geopolymer. The chemical properties of the WA were assessed by studying its chemical composition and the alkalinity of the WA lye produced was assessed through its pH value. The physical and chemical properties of the binders (POFA and GGBS) were assessed using fineness test, specific gravity, chemical composition, loss on ignition as well as mineral morphology. Strength activity index was also carried out to assess the suitability of the binders as pozzolanic materials. Also, the physical properties of TCA such as aggregate crushing value, specific gravity and water absorption were assessed.

The WA lye produced was used as chemical activator mix with sodium silicate to produce an alkaline activator solution with alkaline activator ratio (ratio of Na₂SiO₃ to WA lye) of 1.5 to 3.5 at 0.5 step increment, and a liquid binder ratio (L/B) of 0.45, 0.50 and 0.55, respectively. The fresh and hardened properties of the geopolymer binder using flow table test and compressive strength tests were assessed to come up with the optimum L/B and alkaline activator ratio (AAR).

To improve the properties of the geopolymer binder under ambient curing condition, GGBS was used to replace the POFA binder using different replacement levels (10% to 40% by weight of binder at 10% increment). Workability measurement, initial and final setting time were used to assess the fresh properties of the produced mortar. Also, compressive strength, flexural strength, water absorption, porosity, intrinsic air permeability, drying shrinkage tests were carried out to assess the mechanical and durability properties of the geopolymer mortar. The microstructure and geopolymer reaction products were assessed using scanning electron microscopy (SEM), and fourier transform infrared analysis (FTIR).

The data obtained from the previous tests were used to produce POFA based lightweight geopolymer concrete using TCA to replace the normal aggregate at different replacement levels (20% to 100% by weight of coarse aggregate at 20% incremental level). The properties of the concrete were assessed by testing their workability, compressive strength, flexural strength, tensile strength, modulus of elasticity (MOE) and ultrasonic pulse velocity (UPV). Density and shrinkage tests were conducted to evaluate their physical properties. Porosity, water absorption, sulphate resistance tests were carried out to assess the durability of the concrete.

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The data obtained from the previous tests were used in the fabrication of the lightweight reinforced geopolymer concrete slab. The slab was investigated based on strength and loading characteristics, stress–strain relationship, crack pattern, and vibration response.

1.7 Thesis outline

The thesis consists of five different chapters to cover the engineering, durability, and structural properties of the ambient cured WA lye activated POFA: GGBS geopolymer concrete incorporating TCA. Starting with chapter one, a brief background studies on the current sustainability issues in the construction industry, the contribution of construction industries towards global warming, and the use of geopolymer technology to mitigate the aforementioned problems. Detailed statement of research problem, significance of the research as well as scope of the research were discussed in this chapter.

Chapter two reviewed the previous research on geopolymer concrete, its constituents, and their effect on the overall physical, mechanical, microstructural and durability properties. It also discussed other factors that influenced these properties. The utilization of industrial by-products in the production of conventional and geopolymer concrete were also reviewed. The chapter also shed more light on the utilization of WA in producing self-activating geopolymer mortar and its effects on the physical, mechanical and durability properties of the mortar. A summary was presented at the end of the chapter which identified the gap of knowledge, challenges of geopolymer mortar/concrete as well as ways of mitigating the challenges identified.

Chapter three presented the detailed research program. The materials used, testing parameters, and testing standards used to study the properties of POFA based lightweight geopolymer concrete were presented and discussed. In addition, the chapter laid out the method for testing the newly produced reinforced geopolymer concrete slab. The method of data collection and analysis was also presented in this chapter.

Chapter four include the detail results and discussions of experimental investigation on the fresh and mechanical performance of the POFA based geopolymer mortar activated with WA lye, and the mixing parameters (L/B and AAR) were optimized, the influence of GGBS inclusion as POFA replacement in POFA geopolymer mortar, and the effects of TCA incorporation as coarse aggregate replacement in the POFA: GGBS lightweight geopolymer concrete. Mechanical properties such as density, compressive strength, flexural strength, tensile strength, modulus of elasticity and UPV were examined. Water absorption, porosity, sulphate resistance, and drying shrinkage of the geopolymer concrete were presented and discussed in this chapter. Also, the chapter presents the result of the structural behavior and vibration response of the reinforced concrete and composite slab. A thorough discussion on the performance of the slabs was presented.

Chapter five presents the general conclusions drawn from the research and recommendations for further studies were presented to further improve on the current knowledge on WA lye utilization in geopolymer concrete as an alkaline activator.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

A review on the emergence of geopolymer concrete was presented and discussed in this chapter. The constituents of geopolymer concrete, factors influencing the strength and other properties of the geopolymer concrete were discussed. The influence of chemical activators, curing method as well as effect of particle size of binders on geopolymer mortar/concrete were also reviewed. Other aspects reviewed were the utilization of WA as a source of alkali in self-activating geopolymer, the use of WA as an alkaline activator and their effects on both mechanical and durability properties of geopolymer mortar/concrete. Furthermore, the incorporation of lightweight aggregates in the production of concrete as coarse aggregates and their effect on the mechanical, durability and microstructural properties were also presented and discussed. Also reviewed, were the structural properties of precast lightweight slab. At the end of the chapter, summary was made on the existing gap and the forecasted challenges that may hinder the onsite application of geopolymer mortar/concrete.

2.2 Geopolymer concrete

Concrete is the second most used material on earth after water. It was estimated that 25 billion tons of concrete are produced annually. The huge demand of concrete in the world called for the excessive demand of cement to be used in the concrete production. The production of cement leads to the release of CO_2 in to the atmosphere which is harmful to our environment, it was estimated that for each ton

of cement produced its equivalent amount of CO_2 is released to the atmosphere as greenhouse gas (Shashikant & Prince Arulraj, 2019). This attracts the attention of researchers worldwide to find an alternative to OPC which will be less harmful and environmentally friendly, as such the technology of geopolymer cement emerged.

Geopolymer concrete is produced from waste aluminosilicate materials such as GGBS, FA, rice husk ash (RHA), POFA etc. activated with alkaline activator and is cement free (Shashikant & Prince Arulraj, 2019). The benefit of geopolymers is not only limited to eradication of CO₂ emissions related with OPC production, but also possessed high strength and durability performance better than the OPC concrete (Soutsos et al., 2016). One of the major advantages of geopolymer binders over ordinary Portland cement is that OPC reacts with water forms C-S-H gel, on the other hand that of geopolymer form aluminosilicate gel (Bellum et al., 2020). According to Soutsos et al. (2016), the nature of reactions in repolymerization can be summarized in to the following three stages:

- i. Dissolution of the aluminosilicate solids: the solution of silicate, aluminate and aluminosilicate species are formed by dissolving the aluminosilicates in the pozzolanic solid by alkaline hydrolysis in the high pH solution of the concentrated alkaline activator.
- ii. Gel formation: The dissolution releases some species which are in aqueous phase, containing silicate present from the alkaline activator. A gel is then formed from the supersaturated aluminosilicate solution. At this stage, water is released which resides in pores.
- iii. Polycondensation: the gel species forms a larger network by rearranging and reorganizing. This result in three-dimensional aluminosilicate network of the geopolymer binder.

2.3 Constituent materials

According to Assi et al. (2020), geopolymer concrete consists of three major components, the aluminosilicate binder material such as FA, GGBS, which are the source of aluminosilicate compounds, aggregates which include both fine and coarse and the alkaline activator solution which is mostly a mixture of NaOH, Na₂SiO₃, and water. The following sub-sections elaborate a review on constituent materials of geopolymer concrete.

2.3.1 Alkaline activator

The main process of geopolymerization in the technology of geopolymer cement is the dissolution of silicon and aluminium present in the binder material to form the geopolymer paste. This silicon and aluminium are activated by alkali activators, mostly NaOH and Na₂SiO₃ solution are the most widely used alkaline activators.

It was reported that the composition and concentration of alkaline activators played a major role in the strength development of geopolymer concrete (Ibrahim et al., 2019). The further reported that the compressive strength of natural pozzolana based geopolymer concrete increase with an increase in the concentration of NaOH with about 24% in the compressive strength when the molarity of sodium hydroxide was increased to 14M from 8M. Also, in their research, Kaur et al. (2018) revealed that the compressive strength of geopolymer concrete is directly proportional to the concentration of alkali activator solution as well as AAR, they recorded a maximum compressive strength of 39.95MPa at 28 days curing period from 0.7 AAR and 14M concentration specimen. They further concluded that the compressive strength of geopolymer concrete is directly proportional to molarity and AAR.

Many researchers employed the use of these alkaline activators in their studies i.e. sodium hydroxide and sodium silicate either individually or the combination of both, and best results in both fresh and hardened properties of geopolymer concrete were reported to be achieved when combining the two. Ibrahim et al. (2019) utilizes the combination of industrial grade Na₂SiO₃ solution with NaOH solution of different molarities 8M, 10M and 12M, and a maximum compressive was recorded using a 14M sodium hydroxide concentration and Na₂SiO₃/NaOH ratio of 2.5. In a study on the early strength of NaOH activated FA based geopolymer by Yahya et al. (2017) NaOH of 14M was used to produce a geopolymer and a 7 days maximum compressive strength of 7.1MPa was recorded when NaOH was used alone, as well as 33.33MPa 7 days compressive strength when both NaOH and Na₂SiO₃ were used with NaOH 12M concentration. Similar studies reported that when NaOH was used alone lower strength was achieved at an ambient curing temperature (Phoo-Ngernkham et al., 2015). They further noted that the use of NaOH and Na₂SiO₃ produced a crystalline and amorphous gel which led to the overall strength development. In another study, it was revealed that samples prepared using Na₂SiO₃ alone possessed a strength of 62.9MPa (Huseien et al., 2016). They noted that the strength increase was attributed to production of extra silicate in the system when Na_2SiO_3 was used which accelerate the geopolymeric process. However, they noted that when Na₂SiO₃ alone was used, the workability of the concrete is affected due to high viscosity of sodium silicate. According to Sedira & Castro-Gomes (2020) the combination of NaOH and Na₂SiO₃ as alkaline activator provide a lower value of average pore diameter as a result of more gel formation. Yahya et al. (2017) also reported that alkaline solution mixture of NaOH and Na₂SiO₃ with NaOH concentration of 12M possessed lower water absorption of 2.3% compared to 8M

concentration with 2.48%. It was observed that the combination of both the NaOH and Na₂SiO₃ as alkaline activators produced geopolymers with good workability and strength properties.

2.3.2 Aggregates

Just like conventional concrete, geopolymer concrete uses aggregates inform of fine and coarse for its production, these aggregates serve as body fillers. Coarse aggregates are generally aggregating with particle sizes above 4.75mm and fine aggregates are those aggregates having particle size below 4.75mm.

Most of the coarse aggregates are crushed from naturally occurring rocks which make them to be non-greener construction materials, because their production exhausted our non-renewable natural resources. To comply with the campaign of greener environment, most researchers focused on sourcing other materials that can be used in place of conventional aggregates. Some of the materials identified performed excellently and even possessed the advantage of being lighter than the conventional aggregates as such named lightweight aggregates (LWA). According to ASTM C331-04 (2002) aggregates meeting the requirements of bulk density less than 1120 kg/m³ for fine aggregate and less than 880 kg/m³ for coarse aggregate are classified as LWA, this include aggregates prepared by expanding, pelletizing, or sintering products such as GGBS, clay, diatomite, FA, shale, or slate; aggregates prepared by processing natural materials such as pumice, scoria, or tuff; aggregates derived from and products of coal combustion (Murray, 2007) and those derived from wood waste or palm products combustion such as POCA and TCA. The body of literature on TCA is very limited, but the study reviewed some literature on similar LWA which is POCA.

Palm oil clinker (POC) is a waste generated from this oil mills and generally disposed in an open fields causing health and environmental problems (Tonduba et al., 2019). Many efforts were made on converting this waste material into valuable resources especially in construction industries. Muthusamy et al. (2019) reported the effective utilization of POCA and POFA in the production of high strength concrete having a compressive strength above 80MPa at 10% Ordinary Portland Cement replacement with POFA. Malkawi et al. (2020) also reported that concrete with 100% POCA produced a compressive strength of more than 30MPa and a density of 1821Kg/m³ at 28 days maturity period. However, they reported a reduction in the slump of the concrete which was attributed to the higher water absorption of POCA when compared with normal granite aggregate. In their research, Abutaha et al., (2017) reported that POC powder can effectively be used to coat the surface voids of POCA, which greatly reduced the negative effects on the fresh and hardened properties of the concrete. Nazreen et al. (2018) studied the characterization of lightweight concrete made with POCA, they noted that POCA concrete falls within the lightweight concrete category, having a density of 1990.33kg/m³, this is less than the normal concrete density of 2400kg/m³. They further reported that the compressive strength of 50% and 100% POCA replacement was comparable to control concrete with 100% natural aggregate. They observed same trend in both flexural and splitting tensile strength, respectively. However, it was reported that about 65% loss in compressive strength of POCA specimen with 100% in pervious concrete, although at 25% replacement POC exhibited superior performance, and hence they recommended 25% as an optimum content of POCA in pervious concrete.

The results of these previous researches concluded that POCA can best be utilized in the production of lightweight concrete with the benefits of having better heat insulation, sound absorption and low-density construction materials. Table 2.1 below show some physical properties of POCA as reviewed from previous literatures.

| Reference | Agg. Size (mm) | Specific gravity SSD | Agg. crushing value (%) | Bulk density (Kg/m ³) | Water absorption (%) | Moisture content (%) | Fineness modulus |
|--|----------------------|----------------------------|----------------------------------|---|----------------------------|----------------------------|---------------------|
| Nazreen et al., (2018) | - | 1.92 | - | 817 | - | 1.30 | 2.6 |
| Abutaha et al., (2017) | 4.75-14 | 1.73 | 56.44 | 732 | 3±2 | 1 ±0.5 | - |
| Ibrahim & Abdul Razak, (2016) | 4.75-9.5 | 1.88 | 56.44 | 732 | 3 <u>±</u> 2 | - | - |
| Huda et al., (2018) | 5-12.5 | 1.82 | - | 781.08 | 4.35 | - | 6.75 |
| Malkawi et al., (2020) | 5-14.0 | 1.62 | 18.04 | 823 | 4.43 | - | - |

Table 2.1 Physical properties of clinker aggregate as reported in literature

2.3.3 Aluminosilicate binder

The binder materials in geopolymer concrete can either be industrial or agricultural wastes or even natural raw materials rich in aluminosilicate composition. Geopolymerization process involved the dissolution of Si and Al in alkaline solution, therefore, all pozzolanic materials rich in silica (Si) and alumina (Al) composition can be used as a binder material in geopolymer concrete. ASTM C618 (2010) has classified pozzolanic material into three different classes according to the percentage by weight of silicon oxide (SiO₂), aluminum oxide (Al₂O₃) and iron oxide (Fe₂O₃) as shown in the Table 2.2. Some of these materials include WA, POFA, GGBS, steel slag, metakaolin, RHA and so on.

| Chemical Requirement | | | | | | | |
|---|------|-------|-----|--|--|--|--|
| | | Class | | | | | |
| - | Ν | F | С | | | | |
| $SiO_2 + Al_2O_3 + Fe_2O_3$ min. % | 70.0 | 70.0 | 50 | | | | |
| Sulfur trioxide (SO ₃), max. %. | 4.0 | 5.0 | 5.0 | | | | |
| Moisture content, max. % | 3.0 | 3.0 | 3.0 | | | | |
| Loss on ignition, max. % | 10.0 | 6.0 | 6.0 | | | | |
| Physical Requirement | | | | | | | |
| | Ν | F | С | | | | |
| Fineness: Amount retain when wet-sieved on 45µm sieve, max. % | 34 | 34 | 34 | | | | |
| Strength Activity Index: With OPC at both 7 and 28 days, min. percentage of control | 75 | 75 | 75 | | | | |

Table 2.2 Chemical and physical requirements of pozzolanic materials (ASTM C618, 2010)

Note: Class N and F pozzolana are pozzolanas with minimum 70% of SiO+ Al_2O_3+ Fe₂O₃, and class C are pozzolanas with minimum 50% and less than 70% of SiO+ Al_2O_3+ Fe₂O₃

2.4 Wood ash

WA mostly is a by-product of timber processing industries which came in form of ash and clinkers. According to Tamanna et al. (2020), WA is produced mostly through combustion of wood products in power plants, paper mills, sawmills and other wood-consuming related sectors. This process produced significant amount of WA which was estimated to be around 5% to 15% of the total amount of the processed biomass (James et al., 2012). Although these wastes came from wood source, but several factors may affect their quality and chemical composition as well. Some of these factors were highlighted by Cheah & Ramli (2011) which include the temperature used during the combustion process, types and hydrodynamics of the furnace as well as the species of the mother tree of the wood. They further noted that the pozzolanic oxide compounds of WA varies from different species of the trees. Also, with the classification of wood, it was noted that hardwood produce more ash than softwood (Siddique, 2012).

Serafimova et al. (2011), in a study on the characteristics of WA, revealed that the WA is alkaline in nature with a pH of aqueous extract of 12.6. The plain microscopic observation of the WA shows the dominance of crystalline phase with similar optical characteristics corresponding to calcite. They also observed the existence of fine crystals with $1 - 2 \mu m$ dimension. The study further revealed the particle size of the WA as mostly nano size due to their fine dispersion nature, also the x-ray diffraction analysis revealed the basic crystal forms of the WA as calcium carbonate (CaCO₃), SiO₂ and fairchildite (K₂Ca(CO₃)₂) and highest loss on ignition (LOI) value of 19.6% was determined, which shows the high content of unburned carbon present in the ash.

Grau et al. (2015), reported some physical properties of the WA in their recent studies, which include fineness, specific surface area and specific gravity of the WA used in the study. The WA was reported to include about 25.4% of fine particles of less than 75 μ m, specific surface area, specific gravity, and pH value of 12,025 – 14,025 m²/kg, 2.5 and 12.57, respectively. The specific surface area was high due to a high degree of particle shape irregularity and surface porosity as opined by Cheah & Ramli (2011). The scanning electron microscopy (SEM) analysis of the WA revealed its shape as sub-angular shape with particle size ranging from 10 - 200 μ m (Grau et al., 2015). In a similar research by Rahul Rollakanti et al. (2020) where an uncontrolled burnt WA was used. The WA was obtained by burning wood in an open place, collected, and sieved through 75 μ m sieve. The fineness and specific gravity of the WA was found to be 5.60% and 2.96, respectively.

Chowdhury et al. (2014) reported the chemical compositions of WA used in their study containing major composition of silicon oxide of 65.30%, 4.25% alumina and 2.24 ferric, the total SiO₂, Al₂O₃ and Fe₂O₃ was 71.79% which conform with the minimum 70% of class F and N pozzolana as classified in ASTM C618 (2010). However, other researchers reported the major composition of the WA as calcium oxide with little composition of SiO_2 , Al_2O_3 and Fe_2O_3 , according to their reports the WA didn't meet the minimum requirements of ASTM C618 (2010), but was seen to possess a high percentage of calcium within the range of 44.81% to 61.0%, this classified it as high calcium WA (Abdulkareem et al., 2018; Ban, 2011; Fusade et al., 2019; Hassan et al., 2019). Other researchers such as De Rossi et al. (2020); Eliche-Quesada et al. (2017) and Jindal & Sharma (2020) reported the chemical composition of WA with the total SiO₂, Al_2O_3 and Fe_2O_3 of 52.5%, 57.8% and 58.77% respectively, thus classifying the wood ash as class C according to ASTM C618 (2010). In most of the findings of all the researches, it is noted that wood ash contain a significant amount of potassium oxide (K₂O) of about 12.0 and 14.5, this shows the high alkalinity of wood ash (Arunkumar et al., 2020; Ban and Ramli, 2011). Table 2.3 below presents the different compositions of the WA as reported in various literatures.

| Reference | SiO ₂ | Al_2O_3 | Fe ₂ O ₃ | CaO | MgO | K ₂ O | Na ₂ O | SO_3 | P_2O_5 | LOI |
|----------------|------------------|-----------|--------------------------------|------|------|------------------|-------------------|--------|----------|------|
| Cheah & Ramli, | 2.7 | 1.3 | 1.35 | 61.0 | 8.7 | 12.0 | - | 2.8 | 2.7 | 18.0 |
| (2011) | | | | | | | | | | |
| Ramos et al., | 73.0 | 11.93 | 3.38 | 2.64 | 1.03 | 4.14 | 0.99 | 0.05 | 0.59 | 1.47 |
| (2013) | | | | | | | | | | |
| Chowdhury et | 65.3 | 4.25 | 2.24 | 9.98 | 5.32 | 1.9 | 2.6 | - | - | 4.67 |
| al., (2014) | | | | | | | | | | |
| Eliche-Quesada | 48.6 | 5.94 | 3.26 | 18.1 | 3.2 | 1.85 | 0.92 | 0.14 | 0.52 | 15.6 |
| et al., (2017) | | | | | | | | | | |
| | | | | | | | | | | |

Table 2.3 Chemical compositions of wood ash as reported in literatures

| Abdulkareem et | 2.70 | 1.30 | 1.30 | 61.0 | 8.70 | 12.0 | 2.8 | 2.70 | - | 18.0 |
|------------------|------|-------|------|------|------|------|------|------|------|------|
| al., (2018) | | | | | | | | | | |
| Hassan et al., | 0.98 | 1.51 | 1.19 | 44.8 | 3.81 | 6.89 | 1.64 | 1.07 | 1.72 | 35.9 |
| (2019) | | | | | | | | | | |
| Fusade et al., | 9.34 | 1.18 | 1.98 | 49 | 2.66 | 25.8 | - | 1.02 | 3.29 | - |
| (2019) | | | | | | | | | | |
| De Rossi et al., | 34 | 13.5 | 5.0 | 16.5 | 3.1 | 5.5 | 1.5 | - | - | 14.3 |
| (2020) | | | | | | | | | | |
| Arunkumar et | 7.32 | 0.4 | - | 2.73 | 2.89 | 14.5 | - | - | 2.88 | - |
| al., (2020) | | | | | | | | | | |
| Rahul | 38.3 | 12.87 | 7.64 | 14.3 | 1.94 | 3.28 | - | - | - | - |
| Rollakanti et | | | | | | | | | | |
| al., (2020) | | | | | | | | | | |
| | | | | | | | | | | |

Table 2.3 Chemical compositions of wood ash as reported in literatures (Cont'd)

2.5 Effect of wood ash on conventional concrete

The effect on WA inclusion in conventional concrete will be presented in the following sub-sections.

2.5.1 Fresh properties

Cheah (2011) carried out research to study the physical and chemical properties of high calcium WA and densified silica fume cement. It was observed that the increase in the percentage of the WA led to the increase of water demand of the cement paste to achieve a standard consistency, it was also noticed that the inclusion of WA retards the setting time of the mix. Similar results was observed by Yang et al. (2016) were WA was used to replace cement up to 30% replacement level at a 10% increment. They observed that the increment in the percentage of the WA led to the slightly reduction in the slump of the concrete from 108 - 101.6mm for 10 - 30% replacement level compared to 127mm for control samples. They also noted a delay in setting time of the concrete as the percentage replacement increased. Same effect was noted in the concrete mixture blended with 40% slag. It was also

reported that WA inclusion in foam concrete as partial cement replacement increases the water demand of the mixture as the percentage dosage increased. Similarly a reduction in the density of the mixture was noted (Stolz et al., 2019). Fluidized bed combustion of wood and peat was used by Rissanen et al. (2019) to partially replace cement in mortar at 10, 20 and 40% replacement levels. The outcome of the research indicated that the fluidized bed combustion fly ash (FBCFA) mortar required high dosage of super plasticizer to achieve similar workability with control mortar. In a recent study by Carević et al. (2020), loss of workability was observed with an increase in percentage of WA, this led for addition of mixing water with an average of 10% for cement paste. Similar trend was observed in the cement mortar, the decrease in the degree of wetness of the concrete was 8, 16 and 32% for 5, 10 and 15% WA content. They also noticed a loss in workability of 19, 38 and 44% compared to the control mortar. This finding was proved by the work of Hamid & Rafiq, (2020) were the WA was reported to absorb the mixing water and to maintain same slump with the control specimens, additional plasticizer was increased as the percentage replacement of WA increases.

2.5.2 Mechanical properties

WA was reported to improve the microstructure and mechanical properties of mortar/concrete. Several researchers employed the use of WA as supplementary cementitious material (SCM) in concrete and results obtained shows its ability to improve the concrete quality, hence can be utilized to replace OPC at certain percentage.

Several researchers (Carević et al., 2020; Cheah & Ramli, 2012; Cheah and Ramli, 2011; Fořt et al., 2020; Gabrijel et al., 2021; Garcia & Sousa-Coutinho, 2013;

Rollakanti et al., 2020; Ramos et al., 2013; Ristić et al., 2021; Vijay et al., 2021) reported similar findings which shows that the utilization of WA to partially replace of OPC in mortar/concrete production enhance the compressive strength as compared to 100% cement concrete. The percentage replacement of OPC with WA in most of the studies ranges between 5 - 70% by weight of cement, were 5 - 15% WA were mostly recommended as optimum percentage replacement by most of the researchers. Carević et al. (2020) justified that the strength increase was attributed to the pozzolanic activity of the WA. This reaction occurs between the SiO₂ from the WA and the portlandite [Ca(OH)₂] from the cement during curing period of the concrete.

Contrastingly, other researchers (Bikoko, 2021; Chowdhury et al., 2014; Ghorpade, 2012; Hamid & Rafiq, 2020; Nader et al., 2020; Sigvardsen et al., 2021; Stolz et al., 2019; Thomas et al., 2020) observed that the inclusion of WA in concrete as cement replacement led to lower compressive strength compared to specimens produced with only OPC. In their study Chowdhury et al. (2014), justified that WA performs as a filler in cement medium and not as a binder, this increase the surface area of the filler material required to be bounded by cement.

Cheah and Ramli (2011) tried to improve the property of cement mortar by inclusion of high calcium wood ash (HCWA) and densified silica fume (DSF) to replace OPC. In the study, 0 - 16%HCWA with a fixed 7.5%DSF was used to replace OPC. They observed that 8%HCWA + 7.5%DSF mortar exhibited the highest strength of 54.1MPa at 28 days which was credited to the pozzolanic effect of both HCWA and DSF. Similarly another investigation on the compressive strength of mortar containing HCWA as OPC replacement from 0 - 25% was carried out, they discovered that specimens with 15%HCWA possessed a higher strength at