DEVELOPMENT OF A NEW GREEN ULTRA-HIGH-PERFORMANCE FIBRE-REINFORCED CEMENTITIOUS COMPOSITES

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DEVELOPMENT OF A NEW GREEN ULTRA-HIGH-PERFORMANCE FIBRE-REINFORCED CEMENTITIOUS COMPOSITES

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A project report submitted in partial fulfilment of the requirements for the award of the degree of Bachelor of Engineering (Hons) Civil Engineering (Environmental)

Faculty of Engineering and Green Technology Universiti Tunku Abdul Rahman

January 2024

DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

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Specially dedicated to my beloved grandmother, mother and father

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ABSTRACT

Industries such as oleochemical and water treatment produce a substantial amount of waste byproducts, which include alum sludge ash and glycerine pitch, respectively. In spite of the noteworthy benefits that these industries provide for current development, the waste poses a problem due to the high disposal cost and waste management issues. For that reason, there is a pressing need to develop a financially and environmentally sustainable method of disposing of this waste. This study focused on investigating the feasibility of using waste materials namely alum sludge, and glycerine pitch, in the production of green Ultra-High-Performance Fibre Reinforced Cementitious Composites (UHPFRCC). In this study, alum sludge ash is used to partially replace sand in order to address the issue of sand scarcity that has resulted from overexploitation. Glycerine pitch is used to partially replace plasticiser in order to reduce the high cost associated with the production of UHPFRCC. This approach not only promotes the green practice of reusing waste materials but also ensures the financial and environmental sustainability of UHPFRCC production. This study adopted a phased approach to progressively develop, optimise, and produce the optimal UHPFRCC that strikes a balance between cost, environmental friendliness, and performance. The results from the study indicate that when both alum sludge ash and glycerine pitch are incorporated into the mix, it can enhance the density of the concrete, strength properties of the concrete, and concrete durability. For the replacement of sand with alum

sludge ash and the replacement of plasticiser with glycerine pitch, the optimal levels are 4% and 10%, respectively.

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LIST OF SYMBOLS / ABBREVIATIONS

ст	Centimetre
$^{\circ}C$	Degree Celsius
р	Density, kg/m ³
g	Gram
>	Greater than
kg	Kilogram
kg/m ³	Kilogram per cubic metre
kJ	Kilojoule
kN	Kilonewton
<	Less than
MJ	Megajoule
MPa	Megapascal
m	Metre
m^2	Metre square
m^2/kg	Metre square per kilogram
μm	Micrometre
mm	Millimetre
Ν	Newton
%	Percentage
S	Second

Al (OH) ₃	Aluminium Hydroxide
Al ₂ O ₃	Aluminium Trioxide/ Aluminium Oxide
C_2S	Dicalcium Silicate
C ₃ A	Tricalcium Aluminates
C ₃ S	Tricalcium Aluminates
C ₃ A	Tricalcium Silicates
C-S-H	Calcium Silicate Hydrate
C ₃ A	Tricalcium Aluminates
	Anne in the interference of Material
ASIM	American Society for Testing and Materials
BS EN	British Standard European Norm
EC	Embodied Carbon
EE	Embodied Energy
GHG	Green House Gases
LCA	Life Cycle Assessment
SEM	Scanning Electron Microscopy
	Alum Chuden (new treated)
AS	Alum Sludge (non-treated)
ASA	Alum Sludge Ash
GP	Glycerine Pitch
OPC	Ordinary Portland Cement
S4.75	Sand Size (<1.18mm)
S4.75	Sand Size (<4.75mm)
SI	Silica Fume
STF	Steel Fibre
SCM	Supplementary Cementitious Materials

CHAPTER 1

INTRODUCTION

1.1 Background

Due to extensive urbanisation and population growth, the construction sector faces numerous challenges. Carbon emissions, excessive depletion of natural resources, escalating construction material costs, and structural failure are some of the issues encountered by the current construction industry. As a result, a greater emphasis has been placed on the development of robust, environmentally sustainable, cost-effective construction materials in order to address these problems. A key aspect of this thesis involves the development of cementitious composites that possess superior mechanical properties such as high compressive strength and flexural strength while inflicting minimal damage to the environment.

In recent years, researchers in the field of construction have devoted significant interest to Ultra-High-Performance Fibre Reinforced Cementitious Composites (UHPFRCC) due to their exceptional mechanical properties. In the majority of current literature, it is frequently referred to as Ultra High-Performance Concrete (UHPC). As compared to conventional concrete, UHPC has significantly superior mechanical properties. Kravanja, Mumtaz, and Kravanja (2024) state that UHPC has the potential to attain a compressive strength of up to 120 MPA, a tensile strength of at least 5 MPA, and a flowability of at least 200 mm. The high difference between UHPC and conventional concrete is due to its material composition. For the production of UHPC, the following materials are required: Portland cement, supplementary cementitious materials, well-graded granular materials, a substantial fibre dosage, and an extremely

low water-to-binder ratio of 0.25 (Kravanja, Mumtaz, and Kravanja, 2024). Azmee and Shafiq (2018) further clarified that UHPC with fibre reinforcement can be perceived as a single compound that has been integrated with three innovations in the construction field: self-compacting concrete (SCC), fibre-reinforced concrete (FRC), and high-performance concrete (HPC). Each of the materials within UHPC plays an important role, enhancing certain parts of the mechanical properties and making its overall performance superior and good. Among many materials of UHPC, fibre and binder are the most important. Fibre type and reactive binder are among the main components that affect the mechanical properties of high-performance cementitious composites (Ayim-Mensah & Radosavljevic, 2022). Ductility depends on the type of fibre, whereas compressive strength is determined by the selection of reactive binder (Ayim-Mensah & Radosavljevic, 2022). The control of crack width at the microscale, along with a decrease in brittleness, enhancements in ductility, load-bearing capacity, and energy absorption capacity, can be achieved through the addition of fibres that are randomly distributed within the cementitious matrix (Smarzewski, 2020).

The historical beginnings of UHPC could be found in the 1980s, a time when the product initially became known as a particularly creative construction material after extensive development and research (Kravanja, Mumtaz, and Kravanja, 2024). Such a construction material with superior mechanical properties allows it to be used in some particular infrastructure. Voo, Foster, and Pek (2017) assert that Ultra-High-Performance Concrete (UHPC) has a wide range of applications in many different types of construction, including bridges, architectural features, repair and rehabilitation projects, vertical components such as windmills and utility towers, as well as in the oil and gas sector, offshore buildings, and hydraulic buildings (Azmee and Shafiq, 2018). These structures are usually exposed to adverse conditions or extreme climatic events that could compromise the structure's integrity. Conventional concrete is insufficient for the construction of such applications. However, UHPC's exceptional strength and durability allow it to last long in such conditions, increasing its service life with minimal maintenance and reducing maintenance fees.

1.2 Problem Statements

Carbon emissions from the construction industry have been getting worse and attracting global attention since urbanisation and rapid city development. Cement is manufactured by calcining raw materials, which brings about the generation of a substantial amount of greenhouse gases. According to Aslani and Wang (2019), the process through which the cement is produced is estimated to generate up to 5-7% of global anthropogenic CO₂ emissions.

Year	Billion Metric Tonnes of Carbon Dioxide
1990	2.80
1993	2.85
1996	3.00
2002	3.20
2005	3.40
2008	3.50
2011	3.65
2014	3.70
2017	3.90
2020	4.00

Table 1.1: Global Green House Gases Emissions by Construction Sector from1990 to 2021 (Rivera et al., 2023).

As illustrated in Table 1.1, the construction sector has been a significant contributor to the continuous escalation of global greenhouse gas emissions over the last three decades. The emission is anticipated to peak at 4 billion metric tonnes of CO_2 and to continue to rise consistently over the coming years. This emphasises the significant challenges confronting the construction sector and the urgency to adopt green building practices as an approach of curbing greenhouse gas emissions. In order to minimise carbon dioxide emissions, as a solution, the manufacturing process of concrete should be altered by incorporating supplementary cementitious materials and recycled materials (Aslani and Wang, 2019). According to the research conducted by

Ayim-Mensah and Radosavljevic (2022), they indicate that substituting cement with secondary cementitious materials such as silica fume, fly ash, silica flour, glass powder, and ground granulated blast furnace slag can improve the microstructure and mechanical properties of UHPFRCC. In this study, the proposed solution is to replace cement with silica fume, which can enhance the properties of concrete while reducing carbon emissions.

In addition to carbon emissions, the environment is currently facing a significant challenge in the form of waste management. The environment has been severely impacted by the ever-growing quantity of municipal solid refuse generated as a result of the current era's transition to modernisation and industrialisation (Siddique, 2010). The challenges associated with the safe disposal of municipal solid waste are exacerbated by factors such as the projected growth of waste, the high expenses associated with the operation of landfills, and the lack of available landfills (Siddique, 2010). The environment may be adversely affected by the improper disposal of these solid wastes, which can result in the spread of diseases, the devastation of habitats, visual pollution, and air and water pollution. In addition, certain types of refuse are hazardous, necessitating meticulous management to mitigate public health concerns. For decades, one of the most popular ideas in waste management is the idea to reuse waste in the construction industry, like incorporating it in building material production. Thus, researchers have prioritised the reuse of these waste materials in order to address the issue of waste management, rather than their disposal. If the mechanical properties of UHPC can be improved without compromising its superior mechanical properties by reusing these waste materials, particularly the ones produced in the factory, it would be a big advancement in the construction field. In this study, the proposed solution to this problem is to incorporate waste like alum sludge and glycerine pitch into concrete production since past studies have proved their benefit to concrete. Details on how past studies perform such waste integration into concrete will be discussed in the next chapter. If it is feasible to incorporate this waste into concrete, it will effectively solve the issue related to waste management of this kind, hence reducing the costs associated with disposal and treatment. Recent research on the use of glycerine pitch and alum sludge in the production of building materials has shown positive outcomes. However, there are relatively few historical studies on reusing this waste in ultra-high-performance fibre-reinforced cementitious composites production (UHPFRCC). As a result, it is unknown if these wastes are compatible with the essential components of UHPFRCC, such as steel fibre, silica fume, and high dosages of superplasticizer. Even though they are compatible, it is questionable whether they can achieve ultra-high performance. Therefore, the experimental work in this study initiates with the determination of the compatibility between waste (glycerine pitch and alum sludge) and UHPFRCC essential components.

River sand has been a widely used choice in the production of UHPC. However, in order to safeguard the environment, it is imperative to conduct a wise assessment of the long-term sustainability of the use of river sand. According to Zhang et al. (2018), the reason for this is that the scarcity of river sand has been a significant issue in numerous countries worldwide, including China. This is primarily due to the uncontrolled harvesting and excessive exploitation of these resources from the natural environment for the purpose of major urbanisation and construction (Zhang et al., 2018). The resultant shortages of river sand and overexploitation result in a substantial increase in the price of river sand, as well as damage to the environment (Zhang et al., 2018). For instance, sand mining has the potential to degrade the water quality of natural water bodies, increase the likelihood of soil erosion, and destroy the natural habitat of aquatic species. In order to mitigate the effects of excessive sand mining, many provinces in China, including Fujian, Shanghai, and Zhejiang, have implemented rules and laws that restrict the utilisation of river sand in the building sector Zhang et al. (2018). Consequently, there is a pressing need to identify a substitute for river sand in the production of UHPC. A recent study used alum sludge ash to partially replace fine aggregate in the production of traditional concrete, resulting in improvements in strength and durability. However, replacing fine aggregate with alum sludge ash for ultra-high-performance concrete production remains unexplored. If possible, this practice reduces environmental problems associated with sand exploitation.

Workability is an important requirement for Ultra-High-Performance Concrete (UHPC) to guarantee its high fluidity. This is because UHPC often uses low-to-binder ratios in order to achieve high strength. Therefore, a significant amount of superplasticizer is often used to ensure satisfactory fluidity. However, this results in a significant increase in costs. There is an urgent need to discover an alternative to the

superplasticizer that can maintain the desired workability while reducing the cost. Glycerine pitch serves as a potential alternative due to its zero cost and oily characteristics. However, there have been no published field studies involving the replacement of superplasticiser with glycerine pitch. Therefore, this study attempts to determine the feasibility of such a replacement and, if feasible, identify the optimal extent of the replacement.

1.3 Aims and Objectives

The objectives of the thesis are shown as follows:

- To investigate the feasibility of using waste materials, namely alum sludge and glycerine pitch, in the production of green cementitious composite.
- (ii) To produce ultra-high-performance cementitious composites that are environmentally friendly and economically viable.

1.4 Outline of the Study

This thesis will be divided into 5 chapters. The first chapter contains a brief overview of the concept of Ultra High Fiber Reinforced Cementitious Composites, as well as the problem statement and the objectives that must be accomplished.

In chapter two, the comprehensive concept of ultra high-performance fibrereinforced cementitious composites will be reviewed in detail, including its mechanical properties, composition, production method, and differences from conventional cementitious composites. Subsequently, the source, physical properties, and chemical properties of the materials required for the production of UHPFRCC will be extensively investigated, and the methods by which they have been used in previous research will be discussed.

The next section, which is chapter three, will discuss the materials, apparatus, methodology, and mix proportion required to produce UHFRCC. Various laboratories to evaluate the properties of UHPFRCC will be discussed as well.

Chapter four will look into the results obtained, which will include the manner in which the trend changes in terms of strength, durability, workability, density, and microstructure when each material is added. A life cycle assessment will also be performed to assess the environmental friendliness and cost of the production of UHPFRCC.

The last chapter, which is chapter 5, will conclude the thesis by evaluating whether the objectives in this thesis are achieved and to what extent. It will also provide recommendations to further improve the study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will begin by providing an in-depth discussion of the properties of the materials required for the production of conventional concrete, namely cement, aggregate, and chemical admixture. Next, the concept of Ultra High-Performance Fibre Reinforced Cementitious Composites (UHPFRCC) will be presented, highlighting how it differs from conventional concrete. To produce a new green UHPFRCC, innovative materials like silica fume, steel fibre, alum sludge ash, and glycerine pitch will be used. The properties of these materials will be thoroughly investigated, and a literature review will be conducted to study how previous researchers have employed them to produce UHPFRCC.

2.2 Cement

Cement is the most important ingredient of concrete, as it functions as a binding material due to its adhesive and cohesive properties. When it reacts with water, a paste will form, which functions to bind together the coarse aggregate and fine aggregate. The binding ability of cement is very largely dependent on its physical properties and chemical composition. Therefore, the selection of type of cement is very important in order to obtain desired concrete's properties. The construction industry worldwide offers a wide variety of types of cement, including Ordinary Portland Cement, Portland

Pozzolana Cement, Rapid Hardening Cement, Low Heat Cement, Sulphate Resisting Cement, Blast Furnace Slag Cement, High Alumina Cement and White Cement. The cement types are distinguished by their distinct chemical compositions and characteristics, which enable them to be utilised in a variety of regions and usages.

Bahedh and Jaafar (2018) defined that the fundamental elements of UHPC consist of Portland Cement with high strength, sand with high fineness, a small number of steel fibres, and the absence of coarse aggregates. However, the use of such cement with high strength is not environmentally friendly since it has many environmental impacts.

Cement production is a multifaceted process that generates varying degrees of environmental impact at each stage. In Figure 2.1 below, Yang et al. (2017) illustrate the cement production process, which commences with the mixing of raw materials, followed by incineration, grinding, and lastly cement testing and packaging. Air emissions are primarily caused by the incineration and mixing of raw materials, which release harmful gases such as carbon dioxide, NO_x, sulphur dioxide, particulate matter, and heavy metals. The wastewater generated by housekeeping practices, such as cleansing, is the source of water emissions. Prior to emitting gaseous or liquid effluent into the environment, it is necessary to treat these pollutants to meet certain standards or requirements in order to mitigate environmental pollution. Additionally, in addition to the raw materials that are mined from the environment, a substantial quantity of electricity and water are consumed during the cement production process.



Figure 2.1: Cement Manufacturing Process (Yang et al., 2017).

Each grade of cement that is frequently employed, such as 32.5 MPa, 42.5 MPa, and 52.5 MPa, has a varying degree of environmental impact. Yang et al. (2017) conducted a life cycle assessment to analyse the adverse environmental effects of cement grades of 32.5 MPa, 42.5 MPa, and 52.5 MPa, as shown in Table 2.1 below. The findings indicate that the environmental impact of cement grades 32.5 MPa, 42.5 MPa, and 52.5 MPa, and 52.5 MPa, and 52.5 MPa, 42.5 MPa, and 52.5 MPa, and 52.5 MPa is on the rise in terms of energy consumption, air emissions due to road transport, and the consumption of resources such as limestone, gypsum fly ash. In brief, cement grade with higher strength tends to have more environmental impact. In light of the construction industry's elevated carbon emissions, it is imperative to minimise the usage of high-strength cement while discovering environmentally friendly alternatives that can be used to produce concrete without compromising its

exceptional mechanical properties. Silica fume, steel fibre, and industrial waste such as alum sludge and glycerine pitch will be incorporated into cement grade 30 to find out if it can produce ultra-high-performance cementitious composites.

Table 2.1: Life Cycle Assessment to Evaluate the Environmental Impact of Each	h
Kind of Cement (Yang et al., 2017).	

	Type of cement		
	32.5 MPa	32.5 MPa 42.5 MPa	
Resource Consumption			
Limestone	811.32 kg	974.67 kg	1186.86 kg
Gypsum	44.35 kg	50.11 kg	63.29 kg
Energy Consumption			
Electricity	Electricity 55.81 kwh		85.49 kwh
Coal 72.74 kg		100.78 kg	132.07 kg

2.3 Aggregate

Concrete is composed of a significant amount of aggregate in addition to cement paste, which is why it is of paramount importance. Aggregate is defined as granular materials that include sand, gravel, crushed stone, crushed blast furnace slag, or demolition and construction refuse. Aggregate can enhance the volume stability and durability in comparison to hydrated cement paste alone. Aggregate type, size, and grade are also important aspects to take into account when developing a concrete mix proportion, as they can significantly impact the mechanical properties of concrete, including strength, workability, and durability.

Many researchers have proposed that the utilisation of fine aggregates is necessary for the production of ultra-high-performance concrete. According to Pyo, Kim and Lee (2017), to produce UHPC with extraordinary mechanical properties, the most important aspect of the mix design would be to decrease the size of the particles, which will subsequently reduce the pore size and improve particle packing. In consideration of the aforementioned aspect, the UHPC mixture purposefully eliminates coarse aggregate, thereby reducing the interfacial transition zone and enhancing the homogeneity of the material properties, resulting in a high strength of UHPC (Pyo, Kim and Lee, 2017). Although the absence of coarse aggregate in the specially designed mix proportion contributes to its high strength, it has a significant weakness: high autogenous shrinkage and a costly material cost (Pyo, Kim and Lee, 2017). Large expenses for materials are a result of the fact that a greater proportion of the raw material is wasted in the production of finer aggregates, resulting in lower yields from the same quantity of raw material. Consequently, the decision to incorporate an aggregate with a high degree of fineness should be thoroughly assessed in light of these adverse effects. The nominal maximum size aggregate in the UHPC is below 5 mm, as per the ASTM Standard C1856/C1856M-17 (Zhang et al., 2018).

Increasing construction activities have resulted in a global shortage of sand. This emphasises the critical need to discover alternatives for sand in the manufacturing of UHPC. Researchers have been conducting ongoing research to reduce the exploitation of natural resources from the environment for UHPC production by utilising industrial refuse or by-products. For instance, certain researchers investigated the potential of treated alum sludge as an alternative to river sand in the production of UHPC. In the subsequent chapter, the possibility for river sand to be replaced with treated alum sludge and its properties will be reviewed.

Azmee and Shafiq (2018) state that UHPC should be classified as mortar instead of concrete, as some academics have proposed, in truth. This is due to the inclusion of no coarse aggregate. Nevertheless, UHPC's ductility was improved by incorporating steel fibres, which is why the term "concrete" is used to characterize it (Azmee and Shafiq, 2018).

2.4 Chemical Admixture

A chemical admixture is a substance that is incorporated into concrete to either enhance or alter specific properties. Based on their function, there are four primary categories of chemical additives: set retarders, set accelerators, water reducers (plasticisers or superplasticisers), and air-entraining agents.

Of these, superplasticiser or plasticiser is the most frequently employed in ultra-high-performance concrete. Superplasticisers or plasticisers are employed in concrete mixtures to improve the workability of concrete with a low water-to-binder ratio (Li, Yu, and Brouwers, 2017). Upon adding an amount of plasticisers to the fresh concrete mixtures, the plastisiser particles will adhere to the cement particles through adsorption (Li, Yu and Brouwers, 2017). Subsequently, the cement particles separate from one another by opposing their attractive forces with steric and/or electrostatic forces (Li, Yu and Brouwers, 2017). Dispersion of cement particles will result from the separation of the cement particles, which will release the entrapped water and improve the fluidity and workability of concrete mixtures. Consequently, the incorporation of plasticisers enables the preservation of desirable strength in concrete mixtures with a relatively low water-cement ratio, while also enhancing their workability. While plasticiser and superplasticiser work in the same manner to reduce the water-to-binder ratio required, their extent is different. According to Madhusha (2017), plasticisers can decrease the water requirement by 5 to 15%, while superplasticisers may lower it by up to 30%.

Despite the fact that ultra-high-performance concrete has favourable mechanical properties that enable its application in specific conditions, it has a critical drawback. The widespread use of UHPC is restricted by the high production cost and the extensive energy consumption required during its manufacturing. Additionally, the consumption of a significant quantity of high-quality raw materials is a contributing factor. In particular, superplasticizer is an expensive component of concrete mixtures. Researchers have been making an effort to discover an alternative to superplasticiser that will improve the workability of concrete without compromising its strength. It is possible that glycerine pitch could serve as an alternative to superplasticiser due to its oily properties. More details will be provided in the subsequent chapter.

2.5 Ultra-High Performance Cementitious Composites

According to Zhao et al. (2022), ultra-high-performance concrete (UHPC) is a cementitious composite characterised by its remarkable, strength, toughness, and durability. The compressive strength of UHPC is three to sixteen times that of conventional ones, and its tensile strength exceeds five MPa (Zhao et al. 2022). Energy absorption capacity is another important property of concrete. When exposed to excessive stresses such as impact, concrete with a high energy absorption capacity can undergo controlled deformation without fracturing, as opposed to catastrophic failure that occurs abruptly. UHPC is appropriate for usage in bridges, nuclear power plants, and military facilities where resistance to impact, penetration, and explosion is required due to its high energy absorption capacity under dynamic loading (loading intensities vary over time) (Zhao et al. 2022).

As per Khan, Abbas and Fares (2017) definitions, a cementitious composite is deemed to be a high-performance cementitious composite if at least one of the subsequent attributes is present: 1) favourable workability, 2) high strength and strength gain rate, 3) long-term durability, and 4) low degree of plastic and drying shrinkage. One or more of the subsequent techniques may be employed in the production of UHPC: 1) Reducing the water-binder ratio; 2) Filling the voids in the grain particle distribution; and 3) Implementing advanced methods for mixing, placement, and curing (Khan, Abbas and Fares, 2017). To manufacture UHPC, it is normal for researchers to substitute cement with binary and ternary mixtures of micro-filler minerals, including metakaolin, silica fume, fuel ash, and slag (Khan, Abbas and Fares, 2017). Despite possessing exceptional mechanical properties, UHPC is extremely brittle; this can be remedied through the incorporation of fibres.

In contrast to conventional concrete, UHPC typically comprises a complex composition due to the incorporation of numerous supplementary cementitious composites that serve to improve the material's mechanical properties. In addition, UHPC is an expensive material due to its composition of numerous components. Consequently, researchers have integrated industrial by-products and refuse into cementitious composites in an effort to reduce material costs.

2.6 Difference Between Traditional Concrete and Ultra-High-Performance Concrete

There is a great difference between traditional concrete (TC) and Ultra High-Performance Concrete (UHPC) in terms of composition, mechanical properties, and cost. The detail of the comparison is shown in Table 2.2 below.

	ТС	UHPC		
Mechanical Properties				
~	-			
Strength	Lower	Higher		
	(ranging from 20MPa to 50MPa)	(can exceed 120MPa)		
Tensile Strength	Lower	Higher (with the inclusion of steel fibre)		
Durability	Lower	Higher		
	(can be susceptible to	(less susceptible to		
	chemical attack)	chemical attack and corrosion)		
Ductility and	Lower	Higher		
Toughness				
	Cost			
Material and Production	Lower	Higher (with the inclusion of high- performing additives and steel fibre)		
Need for	Higher	Lower		
Maintenance	(requires crack repairing)	(Higher durability and service life of concrete structure)		
Composition				
	-			
Water-to-binder	Higher	Lower		
ratio	(normally higher than 0.25)	(normally lower than 0.25)		

Table 2.2: Comparison between TC and UHPC.

Cement	Normally grade 30 cements	High-strength cement (grade 50)		
Aggregate	 Coarse aggregate (crushed stone and gravel) Fine aggregate (sand) 	 No coarse aggregate Only fine aggregate (sand) 		
Application				
Applications	General construction like residential areas	Specific conditions like skyscrapers and bridge		

2.7 Silica Fume

Koutný et al. (2018) assert that the incorporation of supplementary cementitious materials to cementitious composites results in the creation of a material with reduced cement content, which has numerous advantageous effects on the mechanical properties of the final material. Researchers have been conducting investigations into the properties of the material by integrating various types of supplementary cementitious materials since they became aware of this. For example, fly ash, slag cement, natural pozzolana, and so on. Silica fume exhibits the most noticeable impact among the numerous supplementary cementitious materials that are available.

Silica fume, also known as micro-silica, is an essential material to produce ultra-high-performance concrete. Silica fume appears as a very fine powder, consisting of mostly SiO₂. According to Raghav et al. (2021), silica fume is a by-product that is generated when high-purity quartz is reduced with coal in electric furnaces throughout the manufacturing process of silicon and ferrosilicon alloys. Additionally, during the manufacturing process of ferrochromium, ferromanganese, ferromagnesium, and calcium silicon alloys, silica fume is accumulated as a byproduct (Raghav et al., 2021). By decreasing carbon emissions and resolving the problem of by-product management, the use of such by-products for construction purposes is green and environmentally beneficial. Also, replacing partially cement with silica fume reduces the overexploitation of limestone, which is the main substance for cement production due to extensive urbanisation.

Silica fume undergoes two types of reactions in concrete: physical and chemical, which enhance the mechanical properties of hardened concrete mass. In the context of physical reactions, silica fume performs micro-filling because of its very fine particulate size. By occupying the spaces between cement granules, micro filling is achieved through the utilisation of silica fume in the form of minuscule particles, which then enhances the matrix packing of cementitious composites (Lou et al., 2023). In the context of chemical reactions, silica fume is a material that is primarily composed of SiO₂ (over 90%), which explains its high pozzolanic properties. Further, C-S-H gel is readily produced when silica fume reacts with calcium hydroxide generated during the hydration of cement (Frýbort et al., 2023). The presence of additional calcium silicate hydrate (CSH) in the concrete not only improves its flexibility but also reinforces its bonds and strengthens its compressive strength (Frýbort et al., 2023). Srinivasan and Sivakumar (1997) further assert that the incorporation of silica fume into cementitious composites results in increased modulus of elasticity, enhanced strength over time, and reduced susceptibility to sodium sulphate attack as a consequence of its low permeability to chloride and water ions (Srinivasan and Sivakumar, 1997). Silica fume is an essential constituent in ultra-highperformance cementitious composites.

A considerable amount of literature has been published on the replacement of cement with silica fume to produce ultra-high-performance concrete. The majority of these studies found that silica fume has a beneficial impact on concrete, resulting in an increase in its compressive strength. For example, Xu et al. (2023) research performed an experiment to investigate the incorporation of silica fume at different ratios in cementitious composites to their compressive strength. The mix proportion of the high-performance cementitious composites is shown in the table below. The results of the experiment demonstrated that when the silica fume is 20%, the maximum compressive strength of UHPC is 130 MPa at the age of 360 days in the condition of 20 Celsius curing, as shown in the figure below. The mix prepared by Xu et al. (2023) will be utilised in this thesis, with a 20% replacement of cement with silica fume. Steel fibre, alum sludge ash, and glycerine pitch will be incorporated subsequently.

Sample	Cement	Silica fume	Natural river sand	water	Superplasticizer*
SF0	1200	0	1100	193.2	21.6
SF5	1140	60	1100	193.2	21.6
SF10	1080	120	1100	193.2	21.6
SF15	1020	180	1100	193.2	21.6
SF20	960	240	1100	193.2	21.6
SF25	900	300	1100	193.2	21.6

 Table 2.3: Mixture Proportion of UHPC (kg/m³) (Xu et al., 2023).



Figure 2.2: Compressive Strength of UHPC under 20 Celsius curing (Xu et al., 2023).

2.8 Alum Sludge Ash

Kaish, Breesem and Abood, (2018) state that alum sludge, which originates from water treatment plants, is generated via a water purification process that employs alum as a coagulant. Tony (2022) stated that in order to guarantee that the water purity is of the highest quality, the water treatment process comprises a sequence of procedures, including screening, coagulation and flocculation, sedimentation, and filtration (see
the treatment flow diagram below). Aluminium sulphate $[A1_2(SO_4)_3.14H_2O]$ is employed as a flocculating agent in the potable water treatment process, resulting in the formation of by-products containing substantial aluminium, known as alum sludge (Tony 2022). The alum sludge that is produced comprises both water and solids, with a water content that varies between 99% (before thickening) and 95% (after thickening) (Tony, 2022).



Figure 2.3: Process Flow of Water Treatment Process.

Each year, water treatment facilities in Malaysia produce over 2 million tonnes of alum sludge (Kaish, Breesem and Abood, 2018). The annual production of this type of alum sludge is going to stay elevated, requiring a significant quantity of open space for disposal in Malaysia (Kaish, Breesem and Abood, 2018). Malaysia has been experiencing a gradual scarcity of land as a result of urbanisation and development; the requirement for land for refuse disposal exacerbates this problem. This type of hazardous waste has the potential to inflict damage to the surrounding environment. As a result, scholars have been endeavouring to identify substitute methods for the disposal of alum sludge in an effort to advance sustainable waste management strategies.

Many scholars have been interested in the research on the use of alum sludge as construction materials. The physical properties and chemical composition of waste greatly affect its potential for utilizsation as construction materials (Ng et al., 2022). Therefore, not all waste is suitable for this purpose. In its dried state, alum sludge is composed of a variety of components, including minerals, sandy particles, organic matter in little amounts (such as humus, organisms, and algae), aluminium sulphate, and sludge-conditioning polymers (Ng et al., 2022). The cement hydration process may be disrupted by the organic substance in the concrete, which can result in the improper bonding of cement particles, which in turn leads to a weaker concrete. Moreover, the durability and service life of the concrete may be significantly impacted by the porous structure that may result from the degradation of organic substances over time. Therefore, to eliminate the organic content within the alum sludge, it is necessary to process the alum sludge by the calcination process. Therefore, in order to eliminate the organic content in the alum sludge, it is necessary to process the alum sludge through the calcination process. The resultant product is referred to as alum sludge ash. After the alum sludge ash is thermally treated, it will result in the formation of substances such as tricalcium aluminates (C_3A) and tricalcium silicates (C_3S), which often appear in ordinary Portland cement (Ng et al., 2022). The calcination procedure is additionally utilised to optimise the microstructure and improve the pozzolanic activity of alum sludge ash (Ng et al., 2022).

In recent years, there has been an increasing amount of literature on the incorporation of alum sludge ash into concrete production. For example, the utilisation of alum sludge as a substitute for fine aggregate in cementitious composites was investigated by Kaish et al. (2021) in the following proportions: 0%, 5%, 10%, and 15%. Then, several tests were conducted to examine its effect on mechanical properties. An increase in fine aggregate replacement by alum sludge from 0% to 10% improves the density, compressive strength, splitting tensile strength, and flexural strength of concrete, according to research by Kaish et al. (2021). However, an increase in the replacement of fine aggregate with alum sludge to 15% results in a noticeable decline in the aforementioned mechanical properties (Kaish et al., 2021). Additionally, a water absorption test was performed on the concrete to assess its durability. It was discovered that water absorption decreases from 0% to 10% but is highest at 15% in comparison to the preceding three proportions (Kaish et al., 2021). This indicates that alum sludge has the potential to reduce the absorption, deterioration susceptibility, and increase the durability of concrete. Kaish et al. (2021) justified that alum sludge ash has a positive impact on concrete's various properties because it performs micro filling and possesses pozzolanic activity.

Incorporation of alum sludge ash to produce UHPC can be a feasible option because the above research has proved that it can improve the mechanical properties of concrete. With natural river sand becoming more and more scarce in the ecosystem, alum sludge ash may be an alternative to replace the fine aggregate in the development of UHPC.

2.9 Glycerine Pitch

A viscous gel with extremely alkali properties and a pH greater than 10, glycerine pitch typically manifests as brown, black, or a combination of the two; however, its colour and consistency are said to change among plants (Hazimah, Ooi and Salmiah, 2003). This substance is predominantly made up of inorganic salts, glycerol, diglycerol, and fatty acids (Hazimah, Ooi and Salmiah, 2003).

Industrialisation is becoming increasingly widespread as a result of the growing demand for commercial products. The generation of waste is an inevitable consequence of the rapid expansion of industry, which poses challenges for waste management. Glycerine pitch is one of the wastes generated in the industry. According to Teoh et al. (2021), refined glycerine, which can also be referred to as glycerol, is a product generated from the oleochemical industry that finds extensive use in fields such as pharmaceuticals and medicine, food and beverage, personal care, tobacco, and even more. The substantial market demand for refined glycerine as a commercial product propelled the growth of this oleochemical industry, which generated a substantial amount of hazardous refuse (Teoh et al., 2021). In the oleochemical industry, glycerine pitch is generated as a byproduct during the production process of refined glycerine (Teoh et al., 2021). Waste management problems are exacerbated by the substantial quantities of such waste generated. Armylisas, Hoong and Tuan Ismail (2023) stated that approximately 200 tonnes of glycerine pitch are produced each month in the oleochemical industry of Malaysia.

In Malaysia, the present procedure for the disposal of glycerine pitch waste is to either incinerate it or encapsulate it in a drum before sending it to a landfill (Teoh et al., 2021). Nevertheless, it is imperative to assess the long-term environmental sustainability of this conventional disposal method, particularly in Malaysian countries where landfills are rapidly reaching their capacity. This conventional disposal method should be also assessed in terms of finance in addition to the environmental burden. The process of disposing of glycerine pitch is energy-intensive and results in a high cost, which will ultimately reduce the profitability of the industry and render it financially unsustainable. Armylisas, Hoong and Tuan Ismail (2023) stated that the cost of disposing of the glycerine pitch ranges from RM1500 to RM3500 per tonne. Glycerine pitch typically consists of dust and contaminants, which hinders its potential for practical utilisation (Teoh et al., 2021). Economic limitations render impracticable traditional approaches to recovering useful components from glycerine pitch, like glycerol and fatty acids (Teoh et al., 2021). Consequently, the reuse of waste is a more cost-effective and environmentally friendly alternative to disposal or treatment for this waste.

A large and growing body of literature has investigated the reuse of glycerine for a variety of purposes. However, due to technical obstacles, there are relatively few historical studies on the incorporation of glycerine pitch in concrete production. This includes the distinct chemical composition of glycerine pitch (which contains various dusts and contaminants), making the interaction with other materials in the concrete mix uncertain, affecting the mechanical properties of cementitious composites, such as strength, workability, and permeability. Therefore, thorough investigation and experimentation are necessary to ascertain whether the use of glycerine pitch in concrete production is a viable option.

Ultra-high-performance concrete is characterised by a low water-to-binder ratio, which necessitates a large dosage of superplasticiser to increase the workability of fresh concrete mixes. Glycerine pitch serves as a potential substance to partially replace superplasticiser with their combined effects to further enhance workability. Lima DaSilva et al. (2020) provide justification for the idea that the oily characteristics of glycerine (glycerol) enable it to serve as a lubricant between the inert particles of cement, thereby rendering the concrete more compacted with reduced pore rates, thereby enhancing its resistance and resulting in increased compressive strength. With the high cost of superplasticiser, if it can be replaced with such zero-cost waste, this can reduce the high cost of UHPC production.

2.10 Steel Fibre

Fibres, which are discrete and short components, are distributed uniformly throughout the matrix of concrete. Fibres are essential elements of cementitious composites because they improve the mechanical properties of concrete, including its strength, durability, and resilience, among others. Typical fibres added to the concrete are steel fibres, synthetic fibres, glass fibres, natural fibres, basalt fibres, polymer fibres, and so on. Each of the fibres possesses unique advantages and disadvantages. In order to determine the appropriate fibre for cementitious composites, a number of factors are evaluated. A variety of applications necessitate the use of distinct types of fibres. This is due to the fact that various applications, such as pavements for roads, bridges, and structures, may necessitate distinct fibre properties based on load-bearing capacity, expected lifespan, and exposed environmental conditions. Additionally, fibre should be derived from green sources, which are those that cause minimal environmental harm and are therefore favourable to the environment. There are numerous determinants of a material's greenness, including resource availability, recyclability, carbon footprint, and so forth. Furthermore, cost-effectiveness is an incredibly important consideration when choosing a fibre type. Using fibre with exceptional properties when it is not required may result in wastage and excessive expenditure.

Concrete, as remains typical, demonstrates inadequate tensile and strain capacities, alongside a notable degree of brittleness; to address these deficiencies, the incorporation of steel fibres can be adopted (Abbass, Khan and Mourad, 2018). The primary benefits associated with the incorporation of steel fibres into concrete are the following: impeding the propagation of macrocracks, inhibiting microcracks from enlarging to the macroscopic level, enhancement of ductility and residual strength subsequent to the initiation of the first crack, and substantial toughness (Abbass, Khan and Mourad, 2018). Structural members are required to possess considerable ductility for safety purposes, enabling appropriate measures to be implemented prior to the

structure's collapse. In light of the fact that the tensile strength of concrete amounts to just 8 to 10 percent of its compressive strength, steel fibre reinforcement is required to enhance the ductility and tensile properties of concrete by arresting and bridging formed cracks (Wu, Shi and Khayat, 2019).

Autogenous shrinkage is a form of shrinkage in which water is lost internally due to hydration reactions occurring within the concrete; this causes a reduction in volume. Due to the low water-to-cement ratio and the incorporation of fine particles like silica fume, autonomous shrinkage in ultra-high-performance concrete occurs more strongly than in ordinary concrete (Wu, Shi and Khayat, 2019). Microcracking may occur as a consequence of the tensile stress generated by autonomous shrinkage in UHPC, which subsequently impairs its strength, durability, and operational capabilities (Wu, Shi and Khayat, 2019). The incorporation of steel fibre is crucial in this instance to prevent further shrinkage, as the fibre's high elastic modulus can decrease the size of cracks and postpone their propagation throughout the shrinkage development process (Wu, Shi and Khayat, 2019).

According to Wu, Shi and Khayat (2019), since fibre can prevent cracking growth and increase the system's intrinsic rigidity, the inclusion of fibre into UHPC can result in a strengthening of its mechanical properties. Typically, an increase in fibre content results in an associated improvement in the mechanical properties of concrete, as an increased amount of fibre is present to support the weight of the load. However, when the fibre content increases to a certain limit, it has an adverse impact on the mechanical properties of concrete. According to Wu, Shi and Khayat (2019), excessive amounts of steel fibre integration in cementitious composites can result in compromised mechanical properties of concrete due to decreased workability, fibre cluster problems, and non-uniform fibre dispersion and orientation.

Numerous scholars have conducted extensive research on the use of steel fibre in concrete to enhance its strength. Song and Hwang (2004), for example, conducted research to determine the optimal steel fibre dosage in concrete. They experimented with varying amounts (based on the volume fraction of concrete) of steel fibre, including 0.5%, 1.0%, 1.5%, and 2.0%. They determined that 1.5% of the steel fibre

dosage is optimal where the corresponding mechanical properties like splitting tensile strength and modulus of rupture are the highest as compared to other dosages.

Wu et al. (2016) stated that various parameters like fibre content, fibre and sample size, fibre blending, fibre distribution, and placing methods can have a significant effect on the flexural properties of ultra-high-performance concrete. The three primary configurations of steel fibre are typically classified into straight, hooked ends, and corrugated (Zhang et al., 2020). The mechanical properties of ultra-highperformance concrete (UHPC) can be influenced by the geometry of steel fibres, which in turn affects the bonding properties (Zhang et al., 2020). Wu et al. (2016) studies investigated the different shapes of steel fibre, namely straight, hooked, and corrugated, on the compressive strength and ultimate flexural strength of UHPC. Their findings demonstrated that the UHPC's compressive and ultimate flexural strengths were at their highest for hooked steel fibres at 28 days, followed by corrugated and straight fibres at 1%, 2%, and 3% of fibre content, respectively. They further justified that three components, namely chemical bond, anchorage mechanical force associated with fibre-end, and friction, provide the strength of the bond at the interface between the fibre and matrix. When compared to other fibre configurations, hooked-shaped fibres offer the most effective mechanical interlock (Wu et al., 2016). In order to maximise the mechanical properties of UHPC, hooked steel fibre will be utilised in the development of UHPC.



Figure 2.4: Different Shapes of Steel Fibres.

2.11 A Summary of the Innovative Materials that Have Been Utilized by Past Researchers in the Concrete Production

As detailed in chapters 2.7 (silica fume), 2.8 (alum sludge ash), 2.9 (glycerine pitch), and 2.10 (steel fibre), the following table summarises the past research that has integrated innovative materials into concrete production. The summarisation of this research allows for mix proportion development in this study.

No.	Strategy	Optimal	Key Findings	Researchers
		Replacement		
		Level		
1	Replace cement with silica fume at 0%, 5%, 10%, 15%, 20% and 25%.	20%	Improve compressive strength.	Xu et al. (2023)
2	Replace fine aggregate with oven-dried alum sludge and 300 °C treated alum sludge at 5%, 10% and 15%.	10%	Improve density, and strength (compressive, splitting tensile, flexural).	Kaish et al. (2023)
3	Steel Fibre is added at volume fraction of 0.5%, 1.0%, 1.5% and 2.0%.	1.5% (based on volume fraction)	Improve compressive strength, splitting tensile strength and modulus of rupture.	Song and Hwang (2004)

 Table 2.4: Summarization of Past Research Incorporating Innovative Materials

 into Concrete Production.

2.12 Conclusion

This chapter reviews the key literature regarding the materials required for the production of Ultra-High-Performance Fibre Reinforced Cementitious Composites (UHPFRCC) as well as the distinctive properties of these materials. The base materials

needed to make concrete are cement, sand, water, and plasticising accelerator. To produce UHPFRCC, there are specific requirements on the base materials. Sand sizes less than 5 mm must be utilised to enhance matrix packing. To ensure high strength, a very low water-to-binder ratio (less than 0.25) is used with a large dosage of superplasticiser to compensate for the fluidity loss. Besides that, innovative materials needed for UHPFRCC production include silica fume, steel fibre, alum sludge ash, and glycerine pitch. Silica fume replacing 20% of cement is optimal to achieve the best micro filling and nucleation effects. To improve strength, a steel fibre dosage of 1.5% (based on the volume fraction of concrete) is optimal. Alum sludge ash replacing fine aggregate at 10% is optimal so that the concrete best performs in strength, density, and water absorption. Glycerine pitch possesses oily characteristics, making it potential to replace expensive plasticising accelerators. However, the percentage of glycerine pitch replacing plasticising accelerator is unknown. Thus, trial and error will be used to find out the optimal percentage for replacement.

CHAPTER 3

METHODOLOGY

3.1 Introduction

The first section of this chapter will lay out the materials required for the production of Ultra High-Performance Fibre Reinforced Cementitious Composites (UHPFRCC) and the initial processing of these materials (if any) before their use in the concrete production process. Afterwards, it will discuss the concrete-making procedures, which include the mixing, placing, moulding, compacting, and curing of specimens. At last, the mechanical properties of UHPC will be tested using various laboratory tests.

3.2 Flow Chart of Study

Figure 3.1 below shows the flow chart of the experimental work for the research of the development of green Ultra-High-Performance Fibre-Reinforced Cementitious Composites. The laboratory work in this project was divided into 3 phases, namely phase 1 (initial mix development), phase 2 (optimisation of mix proportion), and phase 3 (comprehensive testing and validation).

The laboratory work began with the preparation of materials, which are base materials and innovative materials. After the preparation of materials, phase 1 was started. The pyramid method was implemented in Phase 1 to progressively introduce innovative materials like silica fume, alum sludge ash, glycerine pitch, and steel fibre

to investigate the impact of these innovative materials on the compressive strength of the concrete. Subsequently, phase 2 began, to optimise the mix proportion with silica fume, alum sludge ash, and glycerine pitch. The compression test was the only test conducted in phases 1 and 2. This is due to the fact that compressive strength is the most significant property of concrete. Consequently, a significant amount of effort will be devoted to increasing the compressive strength. If the compressive strength of concrete meets the requirement, the other properties, such as density and flexural strength, should not deviate significantly from the standard. Phase 3 was the last stage of the experimental work, with the purpose of further investigating the effect of alum sludge ash and glycerine pitch on concrete properties like compressive strength, flexural strength, durability, workability, and density. Also, in phase 3, the mix was further optimised by upgrading from cement grade 30 to cement grade 50.





Figure 3.1: Flow Chart of the Experimental Work in this Study.

3.3 Preparation of Materials

There are 8 materials used in the production of UHPFRCC. The materials used can be divided into 2 categories, namely base materials, and innovative materials. Base materials are traditional materials used in concrete to provide the basic properties of concrete. Base materials include cement, sand, water, and plasticising accelerator. Innovative materials are materials that function as alternatives to replace a portion of the base materials in the concrete, thereby improving its mechanical properties and achieving a green benefit. Innovative materials include silica fume (partially replace cement), alum sludge ash (partially replace sand), glycerine pitch (partially replace plasticising accelerator) and steel fibre.

3.3.1 Ordinary Portland Cement

Two types of cement were used in this study, namely cement grade 30 and cement grade 50. The grade 30 cement used in this study is Castle Portland Composite Cement, CEM Il / B-L 32.5N, produced by the company YTL Corporation Berhad as shown in Figure 3.2 below. The production of this Castle-branded cement is prioritized with regard to environmentally friendly practices. High-quality limestone is used to replace a portion of the clinker in the production of this cement, thereby reducing its carbon footprint. Besides that, this Castle-branded cement is certified to MS ISO 9001, MS ISO 14001, OHSAS 18001, and MS ISO 50001. It has a setting time (initial) of 155 minutes and soundness of 0.8 mm.

The grade 50 cement used in this study is Orang Kuat High Strength Cement, CEM I 42.5N, 52.5N, produced by the company YTL Corporation Berhad as shown in Figure 3.2 below. This Castle-branded cement is also certified to MS ISO 9001, MS ISO 14001, OHSAS 18001, and MS ISO 50001. It has a setting time (initial) of 130 minutes and a soundness of 1.0 mm.



Castle Portland Composite CementOrang Kuat High Strength CementFigure 3.2: Castle Portland Composite Cement.

3.3.2 Silica Fume

Silica fume, also known as microsilica, is the supplementary cementitious material used in this study to produce UHPC. It is a byproduct generated during the production of silicon and ferrosilicon alloys. It has a specific gravity of 2.22 and is less than 1 μ m in diameter. With its ultrafine particulate size and active pozzolanic effect, it can improve the strength of concrete. Silica fume is hygroscopic due to its high surface area and high degree of particulate refinement, which enables it to readily absorb water from the air. It will form clumps or granules when silica fume absorbs water from the air. Consequently, this may influence the uniform distribution of silica fume throughout the fresh concrete mix, leading to specific areas of weakness that may compromise its strength. Therefore, it is necessary to grind and sieve the silica fume powder using a sieve prior to its use. Additionally, to reduce the formation of clumps, it is recommended that the silica fume powder be stored in a dry environment. The test report for this silica fume is shown in Table 3.1. This silica fume's key parameter satisfies multiple requirements, making it suitable for application.

Property	Specification	Test Result	Meeting the		
			Specifications		
SIO_2	>85	98.1	Yes		
Loss of Ignition	<=6.0	1.48	Yes		
(%)					
Specific Surface	>=15	22.1	Yes		
(m^2/g)					
Pozzolanic	>=85	105	Yes		
Activity Index					
(28) (%)					
Chlorine amount	<=0.3	0.01	Yes		
(%)					

 Table 3.1: Physical and Chemical Characteristic of Silica Fume.



Figure 3.3: Silica Fume.

3.3.3 Sand

The collected sand was cleansed to eliminate any impurities, such as organic matter, salt, clay, silt, and so forth, as these impurities will reduce the strength of concrete. Afterwards, the sand was dried up in an oven at 110°C for 24 hours, as the moisture content of the sand will influence the weight and volume measurements in the concrete mix design. Therefore, it is crucial to dry the sand to ensure precise measurement and proportioning of materials. As previously defined in the subchapter on aggregate in Chapter 2, the aggregate size must be less than 5 mm in order to produce ultra-high-performance concrete. Two different sieve sizes are employed: 1.18 mm and 4.75 mm. To ascertain the differences in their effects on the mechanical properties of UHPC, both diameters (<1.18mm and <4.75mm) were employed.



Figure 3.4: Sand.

3.3.4 Alum Sludge

The alum sludge used in this project was obtained from the company KL-Kepong Oleomas Sdn. Bhd, located in Selangor, Malaysia. It was dried in an oven at a temperature of 120°C to eliminate any moisture held within. Subsequently, the alum sludge that had been oven-dried was ground to a smaller size and subsequently sieved using a 1.18 mm sieve. The oven-dried alum sludge was brown in colour, as in Figure

3.5. After that, the alum sludge passing through a 1.18 mm sieve was calcinated in the furnace at 800 degrees Celsius. The resulting product is termed alum sludge ash, which is white in colour (as shown in Figure 3.6).



Figure 3.5: Sieved and Oven-dried Alum Sludge.



Figure 3.6: Alum Sludge Ash.

3.3.5 Plasticizing Accelerator

This study utilises PYEKWISET, a plasticising accelerator that is produced by PYE PRODUCTS (M) SDN. BHD. It is dark brown in colour and has a pleasant smell. It has a specific gravity of 1.30g/ml. It provides advantages like speeding up setting time, increasing compressive and tensile strength, improving workability and plasticity of the mix, reducing the water-cement ratio, and allowing early de-moulding of pre-cast work.



Figure 3.7: PYEKWISET Plasticising Accelerator.

3.3.6 Glycerine Pitch

The glycerine pitch used in this study was obtained from KL-Kepong Oleomas Sdn. Bhd., Selangor, Malaysia. It is a byproduct formed during the production of refined glycerine. This substance is highly viscous and has a very high pH (more than 10). Its appearance is dark brown. It has an ambient density ranging from 1.0 to 1.1 kg/L. Its

chemical composition is 2.87% water, 5.53% fatty acid, 6.92% salt, 80.13% glycerol, and 4.55% of others (like contaminants and dusts).



Figure 3.8: Glycerine Pitch.

3.3.7 Steel Fibre

The steel fibre utilised in this study is the STAHLCON brand. Straight, corrugated, and hooked are the three most prevalent shapes of steel fibre. In comparison to the other two, the hooked-shape steel fibre demonstrates the highest strength, as previously mentioned in Chapter 2. Therefore, hooked shape was used in this study. It has a length of 3.5 cm (from the start of the hook to the end of the hook) and a diameter of 0.667 mm. It has a density of 7850 kg/m³. Steel fibre serves an important purpose in concrete, which is to improve the tensile strength, provide crack control, and improve the strength of concrete. The steel fibre should be stored in an airtight container to prevent the corrosion.



Figure 3.9: Steel Fibre of brand STAHLCON.



Figure 3.10: Hooked Shape Steel Fibre.

3.4 Phase 1, 2 and 3 Mix Proportions

This section provided the mix proportion used to produce different cementitious composites in Phases 1, 2, and 3. Also, the method on how to develop the mix proportions was discussed as well.

3.4.1 Phase 1 Mix Proportions

The objective of Phase 1 is to establish a mix proportion that is compatible with innovative materials like silica fume, alum sludge ash, glycerine pitch and steel fibre. The pyramid method was employed by progressively adding those innovative materials to the base mix. This was carried out to determine the effect of the addition of each innovative material on the compressive strength of UHPFRCC.

The mix proportions for phase 1 laboratory work are shown in Table 3.2 below. These mix proportions are developed based on past researchers incorporating innovative materials like silica fume, alum sludge ash, glycerine pitch and steel fibre (as summarised in Table 2.4).

The first mix proportion is developed based on Xu et al. (2023) study, having an aggregate-binder ratio of 0.92, addition of superplasticizer at 1.8% based on mass fraction of binder and use of sand size lesser than 1.18mm (the details are shown in table 2.8). Xu et al. (2023) study uses a water-to-binder ratio of 0.17, which can lead to a very low workability mix, making the mixing, placing and compacting process difficult. This is primarily due to the fact that only conventional plasticiser was employed, rather than superplasticizer. Consequently, iterative trials were conducted to determine the optimal water-to-binder ratio from 0.17 to 0.18, 0.19, 0.20, and so forth, until the slurry is capable of flowing at 0.28.

For the subsequent mix, silica fume replaces 20% of the cement, as per the optimised proportion from Xu et al. (2023).

Afterward, for subsequent mix, steel fibre will then be added at 0.3% based on the volume fraction of concrete. Song and Hwang (2004) proved that the optimal level of steel fibre addition to concrete is a 1.5% volume fraction. Nevertheless, the workability of fresh concrete mixtures is significantly impacted when this percentage of 1.5% is attempted in the laboratory, as it leads to clustering. 1.5% steel fibre addition may be optimal under certain cases; however, it may not be as effective for other researchers due to the specific conditions in the laboratory and the proportions of the concrete mix. The laboratory is devoid of advanced machinery that is capable of uniformly and efficiently mixing the steel fibre. Consequently, 0.3% steel fibre is implemented for a conservative purpose, as it was observed to possess acceptable workability. Such an adjustment is necessary to ensure the practical feasibility of the mix.

After that, for the subsequent mix, the sand size was adjusted from <1.18mm to <4.75mm. The nominal maximum size of aggregate is 5mm, as stipulated by ASTM for the production of UHPC. Consequently, in order to minimize environmental impact, the 4.75mm size is implemented. The laboratory's sieve with the closest measurement to 5mm is 4.75mm.

Next, for the subsequent mix, alum sludge replaces 2% of sand. Kaish et al. (2023) proved that 10% of fine aggregate replaced by alum sludge is optimal for strength and density improvement. Similarly, when the percentage of 10% was performed, it was observed that the concrete mix appeared very dry, as alum sludge will absorb water, significantly affecting the mixing process. The recommendation of the research does not always work well for the other researcher, possibly due to the exact composition of alum sludge. Therefore, for a conservative purpose, 2% of alum sludge was performed as this mix possesses acceptable workability.

Then, for the subsequent mix, glycerine pitch replaces 100% and 10% of plasticising accelerator. The effect of how much glycerine pitch is added is unknown; therefore, a trial-and-error approach was implemented by replacing 100% and 10% of the superplasticizer with glycerine pitch.

Finally, the last two mix proportions employ non-treated and treated alum sludge, respectively, to evaluate the necessity of treating alum sludge and compare its impact on the compressive strength of concrete.

Mix	Cement Grade 30	Silica Fume (replace cement)	Sand	Alum Sludge (replace sand)	Water	Plasticising Accelerator	Glycerine Pitch (Replace Plasticising Accelerator)	Steel Fibre (0.3% of concrete volume)
Base-S1.18	1200 (100%)	-	1100 (100%)	-	340	21.6 (100%)	-	-
20SI-S1.18	960 (80%)	240 (20%)	1100 (100%)	-	340	21.6 (100%)	-	-
20SI-0.3STF-S1.18	960 (80%)	240 (20%)	1100 (100%)	-	340	21.6 (100%)	-	26.11
20SI-0.3STF-S4.75	960 (80%)	240 (20%)	1100 (100%)	-	340	21.6 (100%)	-	26.11
20SI-2AS-0.3STF-S4.75	960 (80%)	240 (20%)	1078 (98%)	22 (2%)	340	21.6 (100%)	-	26.11
20SI-2AS-100GP-0.3STF-S4.75	960 (80%)	240 (20%)	1078 (98%)	22 (2%)	340	-	21.6 (100%)	26.11
20SI-2AS-10GP-0.3STF-S4.75	960 (80%)	240 (20%)	1100 (100%)	22 (2%)	340	19.44 (90%)	2.16 (10%)	26.11
20SI-2ASA-10GP-0.3STF-S4.75	960 (80%)	240 (20%)	1078 (98%)	22 (2%)	340	19.44 (90%)	2.16 (10%)	26.11

 Table 3.2: Mix Proportions of Different Cementitious Composites Conducted in Phase 1 of Laboratory Work.

Note:

All the cementitious composites in phase 1 were produced using cement grade 30. Base: Standard mix without any replacements.

SI: Silica Fume (the number preceding denotes the percentage replacing cement).

AS: Non-treated Alum Sludge (the number preceding denotes the percentage replacing sand).

ASA: Alum Sludge Ash (the number preceding the percentage replacing sand).

GP: Glycerine Pitch (the number preceding denotes the percentage replacing plasticizing accelerator).

STF: Steel Fibre (the number preceding denotes the percentage of steel fibre).

S4.75: Sand size less than 4.75 mm.

S1.18: Sand size less than 1.18 mm.

Steel Fibre Volume Fraction Calculation:

- For 1% by volume of steel fibre (based on concrete volume), it is equivalent to 78.500 kg steel fibre for every cubic meter of concrete.
- Then, for 0.3% by volume of steel fibre (based on concrete volume), it is equivalent to 23.550 kg steel fibre for every cubic meter of concrete

Taking 20SI-0.3STF-S1.18 as an example:

Concrete mass = 960g + 240g + 1100g + 340g + 21.6g = 2661.6g

To convert it to volume, use the density of concrete (2400 kg/m3):

Concrete volume =
$$2.662kg \times \frac{1m^3}{2400kg} = 1.109 \times 10^{-3}m^3$$

Amount of steel fibre in volume = $1.109 \times 10^{-3} m^3 \times \frac{0.3}{100} = 3.327 \times 10^{-6} m^3$

Amount of steel fibre in mass =
$$3.327 \times 10^{-6} m^3 \times \frac{7850 kg}{m^3} = 26.11g$$

Therefore, to produce concrete mass with 2661.6 g, 26.11 g of steel fibre is needed.

3.4.2 Phase 2 Mix Proportions

In phase 2, the objective is to optimise the mix proportion to obtain the highest possible compressive strength. The Taguchi method is employed to develop nine combination proportions. Dr. Genichi Taguchi, a Japanese scientist, developed the Taguchi method, an approach to statistical analysis that is used to enhance the performance and reliability of a product or process. According to Fraley et al. (n.d.), this method necessitates the use of orthogonal arrays in the experimental design to arrange the parameters that may influence the process and the levels at which they are supposed to vary. The Taguchi method evaluates pairs of combinations rather than all possible combinations of parameters (Fraley et al., n.d.). This can subsequently assist in identifying the factor that has the greatest effect on product quality with the least amount of time and resources (Fraley et al., n.d.). To put it simply, the Taguchi method requires fewer experiments to identify the optimal mix proportions, as it eliminates the necessity of testing all potential combinations.

The 9 mix proportions are developed by utilizing the best-performing mix proportion in phase 1, which was 20SI-2ASA-10GP-0.3STF-S4.75. This mixture uses sand smaller than 4.75 mm, 0.3% of steel fibre dosage is added (based on concrete volume), glycerine pitch replaces 10% of plasticising accelerator, silica fume replaces 20% of cement, and alum sludge ash replaces 2% of sand. In the formulation of nine mixes using the Taguchi method, three factors and their respective levels are employed: silica fume (replace 15%, 20%, and 25% of cement), alum sludge ash (replace 2%, 4%, and 6% of sand), and glycerine pitch (replace 5%, 10%, and 15% of plasticizing accelerator). Despite the fact that prior research has determined that a 20% replacement of cement with silica fume is preferable, the behaviour of silica fume can vary depending on the concrete mix materials and the specific conditions in the laboratory. Consequently, the testing range comprises 15%, 20%, and 25% to further validate the potential for strength development. Phase 1 has established the feasible levels of alum sludge ash and glycerine pitch, which are 2% and 10%, respectively, without negatively impacting the concrete's strength. The second phase is dedicated to determining the optimal level. The following percentages were evaluated for alum sludge ash: 2%, 4%, and 6%. These percentages do not render fresh concrete mixtures

unmixable. Glycerine pitch was conducted at 5%, 10%, and 15% levels. Likewise, these percentages do not significantly affect the workability.

The steel fibre factor was maintained at constant levels for these nine mix proportions, as its optimal level was determined in phase 1 and found to be effective. Additionally, maintaining a constant steel fibre level eliminates one variable, as the addition of more variables to the experiment increases its complexity. Thus, in order to ensure a manageable experiment process, the maximum number of three variables, namely, silica fume, alum sludge ash, and glycerine pitch were used.

As indicated in the result of Phase 1, the larger sand size (<4.75 mm) did not contribute to the further significant development of strength after 7 days. Therefore, a smaller sand size (<1.18 mm) was used in phase 2. However, smaller aggregates will have a higher surface area, necessitating a greater quantity of water to moisten the surface, thereby reducing the workability. The water-to-binder ratio must be increased to maintain the mix's workability and ensure proper hydration of cement powder. The water-to-binder ratio was increased to 0.32. An unchanged water-to-binder ratio and aggregate-to-binder ratio of 0.32 and 0.92 were used for all these 9 mix proportions. Alum sludge ash was used to replace sand instead of using non-treated since alum sludge ash contributes to strength development, as indicated in the result of phase 1. 9 of the mix's proportions developed are shown in Table 3.3 below.

No	Mix	Cement Grade 30	Silica Fume (replace cement)	Sand (<1.18mm)	Alum Sludge Ash (replace sand)	Water	Plasticising Accelerator	Glycerine Pitch (Replace plasticising accelerator)	Steel Fibre (0.3% of concrete volume)
1	15SI-2ASA-5GP-0.3STF- S1.18	1020 (85%)	180 (15%)	1078 (98%)	22 (2%)	380	20.52 (95%)	1.08 (5%)	26.50
2	15SI-4ASA-10GP- 0.3STF-S1.18	1020 (85%)	180 (15%)	1056 (96%)	44 (4%)	380	19.44 (90%)	2.16 (10%)	26.50
3	15SI-6ASA-15GP- 0.3STF-S1.18	1020 (85%)	180 (15%)	1034 (94%)	66 (6%)	380	18.36 (85%)	3.24 (15%)	26.50
4	20SI-2ASA-15GP- 0.3STF-S1.18	960 (80%)	240 (20%)	1078 (98%)	22 (2%)	380	18.36 (85%)	3.24 (15%)	26.50
5	20SI-4ASA-5GP-0.3STF- S1.18	960 (80%)	240 (20%)	1056 (96%)	44 (4%)	380	20.52 (95%)	1.08 (5%)	26.50
6	20SI-6ASA-10GP- 0.3STF-S1.18	960 (80%)	240 (20%)	1034 (94%)	66 (6%)	380	19.44 (100%)	2.16 (10%)	26.50
7	25SI-2ASA-15GP- 0.3STF-S1.18	900 (75%)	300 (25%)	1078 (98%)	22 (2%)	380	18.36 (85%)	3.24 (15%)	26.50
8	25SI-4ASA-5GP-0.3STF- S1.18	900 (75%)	300 (25%)	1056 (96%)	44 (4%)	380	20.52 (95%)	1.08 (5%)	26.50

Table 3.3: Mix Proportions of Different Cementitious Composites Conducted in Phase 2 of Laboratory Work.

9	25SI-6ASA-10GP- 0.3STF-S1.18	900 (75%)	300 (25%)	1034 (94%)	66 (6%)	380	19.44 (90%)	2.16 (10%)	26.50

Note:

•

All the cementitious composites in phase 2 were produced using cement grade 30.

SI: Silica Fume (the number preceding denotes the percentage replacing cement).

ASA: Alum Sludge Ash (the number preceding the percentage replacing sand).

GP: Glycerine Pitch (the number preceding denotes the percentage replacing plasticising accelerator).

STF: Steel Fibre (the number preceding denotes the percentage of steel fibre).

S1.18: Sand size less than 1.18 mm.

3.4.3 Phase 3 Mix Proportions

In phase 3, the objective will be to further analyse how alum sludge ash and glycerine pitch affect the properties of UHPFRCC like strength, durability, workability, and density. This is to determine the feasibility of using the waste to produce UHPFRCC. Additionally, the second objective of this phase is to further improve the strength of the optimal mix from Phase 2 by transitioning from grade 30 to grade 50 of cement.

To conduct a systematic comparison of the impact of the addition of alum sludge, glycerine pitch, and the transition from cement grade 30 to cement grade 50 on the properties of concrete, five mix proportions will be developed. These five mix proportions are developed using the best mix from phase 2, which was found to be 15SI-4ASA-10GP-0.3STF-S1.18.

Phase 3 also used the pyramid addition method, which was identical to phase 1 in that it involved progressively adding glycerine pitch and alum sludge ash to the mix.

In phase 3, full sets of laboratory tests will be conducted like compressive strength, flexural strength, density test, flow table test, and water absorption test. Scanning electron microscopy was also used to analyse the microstructure of these 5 mix proportions.

No	Mix	Cement	Silica Fume (replace cement)	Sand (<1.18mm)	Alum Sludge Ash (replace sand)	Water	Plasticising Accelerator	Glycerine Pitch (Replace plasticising accelerator)	Steel Fibre (0.3% of concrete volume)
1	15SI-0.3STF- S1.18-G30	1020 (85%)	180 (15%)	1100 (100%)	-	380	21.6 (100%)	-	26.50
2	15SI-10GP-0.3STF- S1.18-G30	1020 (85%)	180 (15%)	1100 (100%)	-	380	19.44 (90%)	2.16 (10%)	26.50
3	15SI-4ASA-0.3STF- S1.18-G30	1020 (85%)	180 (15%)	1056 (96%)	44 (4%)	380	21.6 (100%)	-	26.50
4	15SI-4ASA-10GP- 0.3STF-S1.18-G30	1020 (85%)	180 (15%)	1056 (96%)	44 (4%)	380	19.44 (90%)	2.16 (10%)	26.50
5	15SI-4ASA-10GP- 0.3STF-S1.18-G50	1020 (85%)	180 (15%)	1056 (96%)	44 (4%)	380	19.44 (90%)	2.16 (10%)	26.50

Table 3.4: Mix Proportions of Different Cementitious Composites Conducted in Phase 3 of Laboratory Work.

Note:

SI: Silica Fume (the number preceding denotes the percentage replacing cement).

ASA: Alum Sludge Ash (the number preceding the percentage replacing sand).

GP: Glycerine Pitch (the number preceding denotes the percentage replacing plasticizing accelerator).
STF: Steel Fibre (the number preceding denotes the percentage of steel fibre).
S1.18: Sand size less than 1.18 mm.
G30: grade 30 cement.

G50: grade 50 cement

3.5 Preparations of Apparatus

The testing of various parameters was conducted using three different mould shapes in this investigation, namely cube, prism, and cylinder. The dimensions of the cube mould are 50 mm x 50 mm x 50 mm. The dimensions of the prism mould are 160 mm x 40 mm x 40 mm. The dimensions of the cylindrical mould are 45 mm (diameter) x 40 mm (height). The cube mould was used for the compressive strength test. The prism mould was used for the flexural strength test. The cylindrical mould was used for water absorption and density tests.



Figure 3.11: Cubic Mould (left), Prism Mould (middle), and Cylindrical Mould (right).

3.6 Preparation and Moulding of Specimens

To ensure uniform distribution of materials, all of the dry ingredients—cement, silica fume, sand, alum sludge ash, and steel fibre—were mixed in the mixer for two minutes. Before the dry ingredients were combined with the water, the glycerine pitch was added to the water and stirred until it was fully dissolved. Subsequently, the plasticiser was added to the mixture of water and glycerine pitch. Subsequently, the liquid mixture,

which consisted of water, glycerine pitch, and a plasticising accelerator, was added to the dry ingredients that had already been thoroughly combined. The mixture was then further mixed in the mixer for two minutes until a slurry began to form.

Casting and moulding were the next steps. To ease the demoulding process, the internal surface of the mould required oil application before casting. Nevertheless, it was not recommended to use an excessive amount of oil. This is because the following curing process may be impacted by the presence of an excessive amount of oil on the concrete's surface. After oiling the surface, the fresh mixes were cast into mould. The mould was filled with the fresh mixtures by 1/3, and compaction was accomplished manually, as shown in Figure below). The process of manual compaction and filling was repeated until the mould was filled. Finally, the surface of fresh concrete mixes was levelled off to ensure a smooth surface. After 24 hours of concrete mould hardening, demoulding was performed using an air gun as shown in Figure 3.13 below.



Figure 3.12: Manual Compaction of Fresh Concrete Mixes.



Figure 3.13: Air Gun for Demoulding Purpose.

3.7 Curing of Specimens

After demoulding, the concrete specimens were placed in the water storage tank to cure. The process of curing is crucial for concrete to ensure a proper hydration process and strength gain. The curing times were established as seven and twenty-eight days.

3.8 Laboratory Test

The concrete specimens must undergo four types of tests: engineering, durability, workability, and density (as shown in Figure 3.14 below). The laboratory test is designed to evaluate the ability of the Ultra-High-Performance Fibre Reinforced Cementitious Composites to satisfy the standard or requirement. Additionally, it aims

to identify the optimal mix among numerous mixes that achieves a good balance between environmental friendliness, cost, and performance.



Figure 3.14: Type of Laboratory Test to be Performed.

3.8.1 Flow Table Test

A flow table test is a test to evaluate the consistency of highly workable fresh concrete. This test was conducted according to the standard BS EN 1015-3: 1999. The first step was to clean off the surface of the flow table to ensure high accuracy of data. After that, the mould was placed in the middle of the flow table. The mould was half-filled with fresh concrete mixtures, and it was subsequently tamped down 10 times. The filling and tamping steps were repeated for the second time. The mould was elevated vertically with caution to prevent the moulded mix from being disturbed. After that, the concrete mix was subjected to 15 drops. The fresh concrete mix will then expand, and its expanded diameter will be measured in both directions.

$$f = \frac{D_1 + D_2}{2}$$
(3.1)

Where:

f = average expanded diameter

 D_1 = expanded diameter in x-direction

 D_2 = expanded diameter in y-direction


Figure 3.15: Flow Table Apparatus with Fully Filled Mould.

3.8.2 Compressive Strength

To determine the maximum load that concrete can withstand before failure, it is important to conduct a compression test. The test was conducted according to the standards BS EN 12390-3:2002. It was necessary to wipe the surface of the concrete specimens with clothes that were taken out from the curing containers before the compression test. This protects against the slippage caused by the moisture between the specimens and the machine's plate, which could result in inaccurate data. The machine's baseplate was cleaned and removed from any remaining debris. The concrete specimen then was placed in the centre of the machine's baseplate. The machine had been set up with the appropriate parameters, like the specimen's dimensions and loading rate. The specimen was subsequently loaded until it failed, as indicated by the appearance of cracks. The machine will stop subsequently, and the screen will display the maximum loads in kN and its compressive strength in MPa. The strength of the concrete specimens can be also calculated by Equation 3.2 below.

$$fc = \frac{F}{A} \tag{3.2}$$

Where:

- F_c = compressive strength of concrete specimens, MPa
- F = maximum load at which the concrete specimens fail, N
- A = cross-sectional area of concrete specimens, mm²



Figure 3.16: Compression Machine for Compression Test.

3.8.3 Flexural Strength

Flexural strength, also referred to as modulus of rupture, is the highest stress that a material can withstand at its breaking point when it is subjected to bending load. The flexural strength test was conducted according to standard BS EN 12390-5. The concrete specimens used in this test are 40 mm x 40 mm x 160 mm in dimension. Before the test, similarly, the concrete specimens needed to be wiped off of moisture for the highest accuracy of data. The designated software for the material testing on the computer was opened. The settings were then adjusted on the computer like 'Methods', 'Specimens', and 'Information' to suit the type and dimension of my concrete specimens. Then, the concrete specimens were put on the grip with their middle aligned with the hammer. Next, the 'Crosshead' button was pressed to lower the

hammer until it almost touched the middle of the concrete specimens. Subsequently, the 'Run' button was pressed so that the hammer would put the loads on the concrete specimens until it failed. The result, which is the maximum force in unit Newton, will be shown on the screen. The flexural strength can be calculated by Equation 3.3 below.

$$fs = \frac{3FL}{2bd^2} \tag{3.3}$$

Where:

 $f_s = flexural strength, MPa$

F = maximum load at which the specimen fails, N

L = effective span of specimen, mm

b = width of specimen, mm

d = breadth of specimen, mm

3.8.4 Water Absorption

A water absorption test measures the amount of water that can be absorbed by concrete for a specific period, indicating its durability. This water absorption test was performed according to the standard BS 1881-122:2011. Firstly, the concrete specimens were taken out of the curing tanks. Then, the concrete specimens were dried in the oven at $105 \ ^{\circ}C \pm 5 \ ^{\circ}C$ for 24 hours. This is to remove the moisture. After that, the concrete specimens were removed from the oven and allowed to cool down to room temperature. The dry unit weight of concrete specimens was measured and recorded. This dry unit weight was denoted as W_{dry}.

Next, the concrete specimens were completely immersed in water at a depth at which there is $25 \text{ mm} \pm 5 \text{ mm}$ of water above the top surface of the concrete specimens for 30 minutes. This was followed by the removal of the concrete specimens from the water, and their subsequent shaking and drying with a cloth to eliminate any remaining surface water. The saturated unit weight of concrete specimens was measured and recorded. This saturated unit weight was denoted as W_{sat}. Finally, the percentage of water absorption can be calculated by using formula 3.4 below.

Water Absorption (%) =
$$\frac{Wsat - Wdry}{Wdry} \times 100\%$$
 (3.4)

Where:

W_{sat} = saturated unit weight of concrete specimens

W_{dry} = dry unit weight of concrete specimens

3.8.5 Concrete Hardened Density Test

The laboratory test for concrete hardened state density is performed according to the standard BS EN 12390–7-2009. Two parameters, which are mass and volume, are required to calculate the density of concrete specimens. For mass measurement, there are three conditions under which the mass of concrete specimens can be obtained, namely as received, water-saturated, and oven-dried. In this study, oven-dried mass was used because it removed all the moisture content inside and eliminated the variability of mass. Also, oven-dried density is commonly used for technical specifications in many studies. For volume measurement, three methods are available, namely water displacement, actual measurement, and using designated dimensions. Among these three methods, water displacement is the most accurate, as the concrete specimen is not always the same as the designated volume of the mould because it inevitably has pores and flaw. Therefore, water displacement was chosen for volume measurement. Water displacement was performed by using the buoyancy balance in the workshop.

To obtain the oven-dried mass, the concrete specimens were put in the oven for 24 hours at 105 degrees Celsius. After that, the concrete specimens were taken out of the oven and left to cool at room temperature. Then, the oven-dried mass of concrete specimens was measured and recorded. Finally, the density can be calculated by using the obtained mass and volume by using the formula below. The value of density was expressed in the nearest 10 kg/m3.

$$\rho = \frac{m}{v} \tag{3.5}$$

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Where:

 ρ =density of concrete specimens

m(d)=oven-dried mass of concrete specimen

V=volume of concrete specimens (determined by using the water displacement method)

3.9 Scanning Electron Microscope Analysis

Ever since the year 1960s, scanning electron microscopy (SEM) has been used as a tool to analyse the microstructure of concrete (ASTM International, 2022). This test was conducted according to the ASTM C1723-16(2022) standard. The scanning electron microscope (SEM) can generate images of concrete specimens at a scale ranging from low magnification (15x) to high magnification (50,000x) or even higher. The surface of a concrete specimen is scanned by a focused beam of high-energy electrons in SEM. Various signals are generated when these electrons interact with the elements in the specimen, which can be detected and converted into images. The images generated by the SEM can provide detailed information on the surface topography, composition, and other properties of the concrete. SEM is particularly essential in the field of research and development because it allows the researcher to have a thorough understanding of the material's properties and behaviours and then be able to enhance the material's performance. In this study, a magnification factor of (x5000) was utilised.



Figure 3.17: Machine Used to Conduct Scanning Electron Microscopy (SEM) Analysis.

3.10 Life cycle Assessment

Life cycle assessment serves as a tool for cradle-to-grave assessment of the environmental impacts of the development of a product through its life cycle, from the raw material extraction through manufacturing, transportation, and lastly, disposal. In order to mitigate the environmental consequences of UHPFRCC, this necessitates a new green type. Therefore, a life cycle assessment is implemented to assess the environmental impacts associated with the UHPFRCC developed in this study. The environmental footprint of the developed product was assessed in terms of embodied carbon (kgCO₂/kg) and embodied energy (MJ/kg). Economic analysis was also performed to evaluate the economic feasibility of the developed product.

3.11 Summary

A systematic experimental procedure was carried out in this study to investigate the impact of sand size, silica fume, alum sludge ash, glycerine pitch, steel fibre, and cement grade on the properties of ultra-high-performance concrete (UHPC). The methodology involves the preparation of eight materials, each of which serves a critical role and necessitates processing. The next methodology would be mixing, placing, compacting, and curing. The experimental work was performed in 3 phases that allow for step-by-step development of the best-performing mix in terms of performance, cost, and environmental friendliness. To check whether the products meet the requirements of ultra-high-performance fibre-reinforced concrete, various tests like flow table tests, compressive strength, flexural strength, water absorption, and density tests were performed. A life cycle assessment was also performed to evaluate the environmental footprint and cost of the developed product.

CHAPTER 4

RESULT AND DISCUSSIONS

4.1 Introduction

This chapter provided an in-depth assessment of the findings obtained from phases 1, 2, and 3. The objectives of each phase of the study are to develop, optimise, and ultimately validate the Ultra-High-Performance Fibre Reinforced Cementitious Composites. Laboratory work that is conducted in phases can facilitate systematic investigation, which simplifies the identification of trends and correlations and enables precise comparisons between various mixtures. A further advantage of conducting such a multiphase study is to mitigate risks. For example, if the mixes are not performing well or do not satisfy the standard, modifications can still be made before the next resource-intensive phase is implemented. Overall, the multiphase study, which starts with the initial mix development, progresses through mix optimisation, and ends with comprehensive validation, and testing, gives a clear pathway for the development of the final product, a UHPFRCC that effectively balances environmental friendliness, cost, and performance.

4.2 Findings of Phase 1 Laboratory Work (Initial Mix Development)

The compressive strength of the various mix proportions that were conducted in phase 1 is presented in graphical form (Figure 4.1). To demonstrate the reliability of the results acquired, the standard deviation is represented as an error bar. Higher standard deviations indicate the presence of an outlier, which reduces the reliability of the results. Adequate mixing is important for avoiding the emergence of outliers and thereby enhancing the reliability of the data obtained. The homogeneity of the concrete mix throughout its mass is ensured by the efficient mixing process, which avoids the emergence of any outliers in the quantity of concrete that is produced.



Average Compressive Strength of Different Cementitous Composite In Phase 1 _____ 7d ____28d

Figure 4.1: Graph of Average Compressive Strength against Different Cementitious Composites in Phase 1.

The first mix, (Base-S1.18), is a traditional concrete consisting of only cement, sand, water, and plasticising accelerator without the addition of any innovative materials. It achieves compressive strengths of 28.303 and 50.255 at 7 and 28 days of hydration, respectively. There is a significant increase in strength from 7 days of hydration to 28 days of hydration. This suggests that the hydration of cement takes place continually, leading to the production of hydration products such as calcium silicate hydrate (C-S-H) gel, which gradually increases the strength of concrete.

The second mix (20SI-S1.18), compared to the first mix (Base-S1.18), replaces 20% of cement with silica fume. The average compressive strength increases by 46% and 35% at 7d and 28d. This indicates that silica fume has a positive effect on the concrete. As mentioned in the literature review, this can be attributed to the nucleation effect and micro filling function of silica fume. Due to its ultra-fine particulate size, silica fume powder has the ability to fill in the vacant spaces between particles, thereby improving matrix packing and compressive strength. The nucleation effect enables the rapid formation of hydration products, resulting in a greater development of strength in concrete at an early stage. The strength development trend aligns with the findings of Xu et al. (2023). Xu et al. (2023) found that the mix showed a 17% strength increment at 7 days and an 8% strength increment at 28 days when 20% of the cement was replaced with silica fume, in comparison to the control mix. The explanation for the greater strength increment at 7 days as compared to 28 days could be that silica fume and calcium hydroxide react quickly, which speeds up the early strength development.

The third mix (20SI-0.3STF-S1.18), compared to the second mix (20SI-S1.18), adds 0.3% of steel fibre based on the volume fraction of concrete. The average compressive strength increases to 54.456 MPa and 83.098 MPa at 7d and 28d. Such an increment in compressive strength indicates that steel fibre has a positive effect on the concrete. That is, steel fibres provide crack resistance to the concrete by bridging the microcracks, preventing them from transforming into macrocracks, as mentioned in the literature review. Consequently, the concrete is able to withstand an additional amount of load before cracking.

The fourth mix (20SI-0.3STF-S4.75), compared to the third mix (20SI-1STF-S1.18), changes the sand size from 1.18 mm to 4.75 mm. At 7 days, the average compressive strength increased from 54.456 MPa to 57.394 MPa, while it decreased from 83.098 MPa to 65.885 MPa at 28 days. This suggests that the utilisation of larger sand sizes results in a high level of strength at an early age; however, the subsequent development of strength is minimal. Smaller aggregate usually has a smaller interfacial transition zone, which increases the strength, as discussed in the literature review. Besides that, the smaller aggregate size makes the steel fibre inside the concrete less susceptible to corrosion. Yoo, Shin and Banthia (2021) assert that due to its extremely dense microstructure, Ultra High-Performance Concrete is quite resistant to chloride ion penetration (the culprit for steel fibre corrosion). The microstructure becomes less dense as the aggregate size increases, which means that the embedded steel fibre is more susceptible to corrosion. Consequently, the steel fibre is unable to provide crack resistance, resulting in a decrease in the compressive strength of concrete,

despite the continuous hydration of cement to compensate for the loss of strength.

The fifth mix (20SI-2AS-0.3STF-S4.75), compared to the fourth mix (20SI-0.3STF-S4.75), replaces 2% of sand with alum sludge (non-treated). On the seventh day, the compressive strength decreases from 57.394 MPa to 43.040 MPa, and on the 28th day, it decreases from 65.885 MPa to 43.3 MPa. Additionally, it is noticeable that the 28-day strength is nearly identical to the 7-day strength, despite the fact that the average reading was obtained for both days in order to obtain precise data. It is obvious that the concrete's strength is significantly reduced by the untreated alum sludge. As previously mentioned in the literature review section, the untreated alum sediment contains organic substances that disrupt the cement hydration process, thereby reducing the development of strength. However, this outcome is contrary to that of Kaish et al. (2021). Kaish et al. (2021) conducted a research study to determine the feasibility of replacing sand with non-treated alum sludge (oven-dried at 105°C only) and treated alum sludge (heated at 300°C) at 5%, 10%, and 15%, respectively. The results of their research demonstrate that the compressive strength of the sample is significantly higher than that of the control sample in all curing ages when the fine aggregate is replaced by both non-treated and treated alum sludge at 5% to 10%. A possible explanation for this might be that the chemical composition of alum sludge varies from plant to plant. For example, some plants produce alum sludge that contains

a greater amount of organics, which can significantly impede the hydration process of cement.

The sixth mix (20SI-2AS-100GP-0.3STF-S4.75), compared to the fifth mix (20SI-2AS-0.3STF-S4.75), replaces 100% of plasticising accelerator with glycerine pitch. The purpose of comparing the sixth mix with the fifth mix is to determine if glycerine pitch can completely replace the plasticising accelerator. Additionally, this sixth mix includes all the innovative materials (silica fume, alum sludge, glycerine pitch, and steel fibre) to assess their compatibility with one another. The results indicate the 7 day strength is 0.952 MPa and 28 day strength is 3.300 MPa. The significant reduction of compressive strength indicates that 100% replacement of plasticising accelerator with glycerine pitch is not a viable option, as it can have a profound negative effect on concrete. The glycerine pitch has significantly hindered the stiffening and hardening process of concrete, as seen by the low early strength of 0.952 MPa at 7 days. Regarding its impact on long-term strength, the concrete exhibits a notable low strength of 3.300 MPa at 28 days, suggesting that it continues to lack strength even after a long curing period. It is worth mentioning that glycerine pitch is a type of waste produced by industries, whereas plasticising accelerator is a commercial product specifically engineered to enhance the characteristics of concrete. As a result, glycerine pitch may not possess the same favourable characteristics as a plasticising accelerator. Thus, glycerine pitch is not able to replace plasticising accelerator completely.

The seventh mix (20SI-2AS-10GP-0.3STF-S4.75), compared to the fifth mix (20SI-2AS-0.3STF-S4.75), replaces 10% of plasticising accelerator with glycerine pitch. The 7d strength decreased from 46.040 MPa to 40.16 MPa while the 28d strength increased from 46.3 MPa to 51.003 MPa. This suggests that glycerine pitch cannot contribute to high early strength, but it can increase concrete's long-term strength. The reason why the replacement of the plasticising accelerator by glycerine pitch can lead to a reduction of early strength may be because glycerine pitch does not possess plasticising accelerator's distinct function to improve setting time and improve early strength. The increase in long-term strength is due to the fact that glycerine pitch acts as a lubricant between particles, making the concrete more compact, as aligned with the theories in the literature review. The more compacted the concrete, the higher

strength it gains. Reducing early strength while increasing long-term strength may seem like an acceptable trade-off. Besides that, considering that glycerine pitch is a waste, replacing high-cost plasticising accelerators is beneficial in terms of both performance and environmental friendliness. Therefore, rather than replacing the plasticising accelerator entirely, it is better to replace it partially with glycerine pitch.

The eighth mix (20SI-2ASA-10GP-0.3STF-S4.75), compared to the seventh mix (20SI-2AS-10GP-0.3STF-S4.75), the replacement of sand with 2% non-treated alum sludge has been changed to 2% alum sludge ash. The purpose of this comparison between the ninth and eighth mixes is to evaluate the impact of non-treated alum sludge and alum sludge ash on the strength of concrete. The compressive strength increased from 40.16 MPa to 59.841 MPa at 7 days and from 55.643 MPa to 74.390 MPa, as indicated in the result. A significant increase in compressive strength is observed when the non-treated alum sludge is changed with alum sludge ash. Nontreated alum sediment contains certain organic substances that impede the proper hydration of cement. Therefore, it is imperative to subject non-treated alum sludge to the calcination process in order to enhance its pozzolanic activity, eradicate organic substances, and improve its microstructure, as mentioned in the literature review. Alum sludge ash, a byproduct of the calcination process, exhibits excellent pozzolanic activity and can contribute to the development of concrete strength. Additionally, alum sludge ash is inorganic, which ensures that it does not degrade over time and reduce the strength of concrete.

The optimal mix developed in Phase 1 experimental work is the eight mix (20SI-2ASA-10GP-0.3STF-S4.75). This mix is compatible with innovative materials (silica fume, alum sludge ash, glycerine pitch, and steel fibre). Also, it achieves the greatest compressive strength at 7 days among many other mix proportions. Nevertheless, this mixture may be required to enhance the long-term strength by utilising sand with a size of less than 1.18mm instead of less than 4.75mm.

In conclusion, it was proved that incorporation of innovative materials, which are silica fume, steel fibre, alum sludge ash, and glycerine pitch, is able to enhance concrete strength. However, the optimal level at which the innovative materials can work best for the concrete is unknown. As a result, phase 2 of the experimental work aims to determine the optimal level of these innovative materials. Additionally, glycerine pitch can only partially replace plasticising accelerator, but not completely. Additionally, it is beneficial to prevent the incorporation of non-treated alum sludge into UHPFRCC, as the organic substances will undergo degradation. The calcination process must be applied to alum sludge in order to produce UHPFRCC. Smaller sand sizes (<1.18 mm) should be employed instead of larger sand sizes (<4.75 mm) to enhance the long-term strength of UHPFRCC. Therefore, phase 2 of the experimental work will use sand size (<1.18 mm) instead of sand size (<4.75 mm).

4.3 Findings of Phase 2 Laboratory Work (Optimization of Mix proportions)

The average compressive strength of different cementitious composites conducted in Phase 2 experimental work is shown in Figure 4.2 below. To select the optimal mix among the nine mixes, the most important criteria would be performance (strength, in this case), followed by cost, and lastly, environmental friendliness.

First, in terms of strength, the second mix (15SI-4ASA-10GP-0.3STF-S1.18) performs the best compared to the other eight mixes. While the compressive strength of the second mix is almost the same as that of the fifth and seventh mixes at 7 days, it is noticeably higher than them at 28 days.

To produce UHPFRCC with low cost, and most importantly, to reduce the steel fibre dosage because steel fibre is the most expensive material in UHPFRCC production. All the mixes in phase 2 have the same steel fibre dosage, so the second factor will be evaluated. Secondly, it should incorporate the least amount of alum sludge ash. This is because the energy demand associated with the calcination of alum sludge at 800 degrees Celsius is very high, resulting in a high electricity bill. Since the second mix uses a medium amount of alum sludge ash, the cost is acceptable.

To produce UHPFRCC that is environmentally friendly, it must incorporate a large volume of waste to inflict the least negative impacts on the environment. Given that the second mix employs a medium quantity of waste—4% alum sludge ash and

10% glycerine pitch—in comparison to the other eight mixes, the second mix is considered to be quite environmentally friendly.

After considering three factors: cost, environmental friendliness, and performance, the second mix (15SI-4ASA-10GP-0.3STF-S1.18) was determined to be the optimal mix in phase 2. Nevertheless, the compressive strength of the optimal mix in phase 2 is lower than that of the optimal mix in phase 1. The optimal mix in phase 1, 20SI-2ASA-10GP-0.3STF-S4.75, achieves 59.841 MPa at 7 days and 74.39 MPa at 28 days. The optimal mix in phase 2, 15SI-4ASA-10GP-0.3STF-S1.18, achieves 50.526 MPa at 7 days and 65.024 MPa at 28 days. The decrease in strength is a result of the higher water-to-binder ratio in phase 2 compared to phase 1. The water-to-binder ratio in Phase 1 is 0.28, while in Phase 2, it is 0.32. It is necessary to increase the water-to-binder ratio in order to ensure adequate workability, which in turn facilitates mixing and compaction. Therefore, in phase 3, the optimal mix from phase 2 (15SI-4ASA-10GP-0.3STF-S1.18) will be further optimised by transitioning from cement grade 30 to cement grade 50 in order to compensate for the strength loss caused by the increase in water-binder ratio.



Average Compressive Strength of Different Cementitous Composite In Phase 2

Figure 4.2: Compressive Strength of Different Cementitious Composites of Phase 2 at 7d and 28d Age of Hydration.

4.4 Findings of Phase 3 Laboratory Work (Comprehensive Testing and Validation)

Phase 3 of the experimental work develops 5 mix proportions by progressively adding the innovative material to the mix. The purpose is to determine how the addition of alum sludge ash and glycerine pitch affects various concrete's properties like compressive strength, flexural strength, workability, density, water absorption, and porosity. Additionally, the aforementioned properties will be assessed as the concrete transitions from cement grade 30 to 50.

4.4.1 Flow Table Test

For UHPFRCC, workability is a crucial parameter to be determined. The purpose of developing a functional UHPFRCC is to make sure that fresh concrete is easy to move, pump, place, and compact. Inadequate workability of UHPFRCC can result in difficulties on actual construction sites. The densities of 5 different cementitious composites developed in phase 3 experimental work at 7 days and 28 days are shown in Figure 4.3 below.



Workability of Different Cementitious Composites Developed in Phase 3

Figure 4.3: Workability of Different Cementitious Composites in Phase 3.

The first mix, 15SI-0.3STF-S1.18-G30, serves as a control. It achieves a spread diameter of 15.5 cm. The second mix (15SI-10GP-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 10 percent of the plasticising accelerator with glycerine pitch. The spread diameter increases from 15.5 cm to 16.9 cm. The increase in spread diameter (workability) is a result of the combined effect of plasticising accelerator and glycerine pitch, which contribute to the fluidity of fresh concrete mixtures through different mechanisms. the plasticising accelerator functions by releasing the confined water between the granules, while glycerine pitch provides an oily characteristic that acts as a lubricant between granules. When the effects of both plasticising accelerator and glycerine pitch combine, fresh concrete mixes become more workable and more fluid, allowing for better compaction.

The third mix (15SI-4ASA-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 4 percent of the sand with alum sludge ash. The spread diameter decreases from 15.5 cm to 14.1 cm. The decrease in spread diameter (workability) is due to the tendency of alum sludge ash to absorb water, leading to drier fresh concrete mixes. This finding is consistent with that of Kaish et al. (2021),

who found that the workability decreases as the percentage of fine aggregate replaced by treated alum sludge increases. This is indicated by a decrease in the slump value and compacting factor. It was asserted that the reason for this is the water retention behaviour of alum sludge ash, which causes the surface to take in greater amounts of water than a sample that does not contain alum sludge ash (Kaish et al., 2021).

The fourth mix (15SI-4ASA-10GP-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 4 percent of the sand with alum sludge ash and 10 percent of the plasticising accelerator with glycerine pitch. The spread diameter increases slightly from 15.5cm to 15.7 cm. Even though the mixes contain alum sludge ash, which has the ability to absorb water and reduce fluidity, the fluidity loss is compensated for by the combined effect of glycerine pitch and plasticising accelerator.

The fifth mix (15SI-4ASA-10GP-0.3STF-S1.18-G50), as compared to the fourth mix (15SI-4ASA-10GP-0.3STF-S1.18-G30), changes from the use of cement grade 30 to cement grade 50. The spread diameter decreases from 15.7 cm to 15.6 cm. This is because cement grade 50 has a slightly higher fineness than cement grade 30, which has a larger surface area and requires more water to wet the surface. Consequently, when compared to cementitious composites produced of cement grade 30, cementitious composites made of cement grade 50 seem drier and have poorer fluidity and workability at the same water-to-binder ratio.

4.4.2 Compressive Strength

The compressive strength of 5 different cementitious composites at 7 days and 28 days is shown in Figure 4.4 below.



Figure 4.4: Compressive Strength of Different Cementitious Composites in Phase 3.

The first mix, 15SI-0.3STF-S1.18-G30, serves as a control. It achieves a compressive strength of 40.354 MPa and 50.183 MPa at 7 days and 28 days, respectively. The second mix (15SI-10GP-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 10 percent of the plasticising accelerator with glycerine pitch. At 7 days, the compressive strength decreases from 41.354 MPA to 41.146 MPa, and at 28 days, it increases from 50.183 MPa to 61.905 MPa. The manner of strength development is similar to that of phase 1, in which the glycerine pitch results in a decrease in early strength but an increase in long-term strength. Such an increment in long-term compressive strength is due to the combined effects of glycerine pitch and plasticising accelerator, increasing the workability of fresh concrete mixes and making them more compact, achieving higher compressive strength.

The third mix (15SI-4ASA-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 4 percent of the sand with alum sludge ash. Both the early strength and long-term compressive strength increase due to such replacement. This result is somehow similar to that performed by Kaish et al. (2021). Kaish et al. (2021) show that compressive strength increases when the replacement of

fine aggregate with treated alum sludge increases from 0% to 10%. Kaish et al. (2021) justified that alum sludge ash can fill up the internal voids within concrete due to its highly fine particle size. Also, more binder is generated during the pozzolanic reaction, further increasing the strength of the concrete.

The fourth mix (15SI-4ASA-10GP-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 4 percent of the sand with alum sludge ash and 10 percent of the plasticising accelerator with glycerine pitch. The addition of both glycerine pitch and alum sludge ash increases the early strength very slightly, but the long-term strength increases significantly compared to mixes 1, 2, and 3. Glycerine pitch and alum sludge ash work in different ways, but when combined, they can significantly increase compressive strength.

The fifth mix (15SI-4ASA-10GP-0.3STF-S1.18-G50), as compared to the fourth mix (15SI-4ASA-10GP-0.3STF-S1.18-G30), changes from the use of cement grade 30 to cement grade 50. The compressive strength increases significantly at 7 days and 28 days. This is because cement grade 50 contains a greater amount of clinker, which is a key component in the development of strength. Clinker contains the principal compounds C3S and C2S. This assists in the development of high early and long-term strength.

Among the mixes, the fifth one (15SI-4ASA-10GP-0.3STF-S1.18-G50) has the highest compressive strength, making it the optimal mix. Nonetheless, the compressive strength of ultra-high-performance concrete must be greater than 120 MPa. The fifth mix failed to meet the compressive strength criteria set by UHPC. This mix only meets the requirements for High-Performance Concrete, which has a compressive strength between 70 and 80 MPa. To produce UHPC, a water-to-binder ratio of less than 0.25 must be used, but it was found that the mixing and compacting process is not workable with this water amount. To make it workable with this ratio, it should utilise advanced mixing and compacting equipment. Also, superplasticiser needs to be used instead of normal plasticiser. This is the suggestion for key improvement of the compressive strength in this study.

4.4.3 Flexural Strength

Flexural strength is the ability of concrete to resist bending and tensile forces. Flexural strength is also an important property of concrete because many structural components, like beams and slabs, are subject to bending. The flexural strength of 5 different cementitious composites at 7 days and 28 days is shown in Figure below.



Figure 4.5: Flexural Strength of Different Cementitious Composites in Phase 3.

The trend of strength development as a result of the addition of each material is similar to that of compressive strength. Glycerine pitch increases the long-term flexural strength due to better compaction. Further, alum sludge ash increases the flexural strength as well due to pozzolanic activities and internal void-filling effect. This result is also consistent with that performed by Kaish et al. (2021). Kaish et al. (2021) show that flexural strength increases when fine aggregate is replaced with treated alum sludge, from 0% to 10%. Moreover, when it changes from cement grade 30 to grade 50, the flexural strength increases further.

The fifth mix, being the optimal mix, achieved a flexural strength of 16.01 MPa at 28 days. In terms of flexural strength, this mix achieves the requirement of ultrahigh-performance concrete, which has a flexural strength of more than 15 MPa.

4.4.4 Water Absorption

One of the key characteristics of concrete that indicates its durability is its ability to absorb water. Concrete is susceptible to deterioration due to the absorption of numerous adverse chemicals, which reduce its durability. Concrete must therefore absorb as little water as possible to prolong its life and lower maintenance costs. The water absorption of 5 different cementitious composites at 7 days and 28 days is shown in Figure below.



Figure 4.6: Water Absorption of Different Cementitious Composites in Phase 3.

The first mix, 15SI-0.3STF-S1.18-G30, serves as a control. It achieves water absorption of 4.61% and 3.31% at 7 days and 28 days, respectively.

When glycerine pitch replaces 10% of the plasticising accelerator, it makes the concrete more compacted, thereby slightly reducing its tendency to absorb water after 7 and 28 days.

When alum sludge ash replaces 4% of sand, water absorption also decreases. This finding is also consistent with that of Kaish et al. (2021). The findings of Kaish et al. (2021) suggest that water absorption decreases when treated alum sludge replaces fine aggregate from 0% to 10%. They justified the decrease in water absorption by saying that the alum sludge ash performs a micro-filling effect, which fills the internal cavities and pores. However, water absorption increases when it rises from 10% to 15%. The excessive quantity of alum sludge ash added is the cause of the increase in water absorption, as some of it remains unhydrated and has a propensity to absorb water (Kaish et al., 2021).

When both glycerine pitch and alum sludge ash are incorporated, the effect intensifies, making the water absorption lower.

As the cement grade transitions from 30 to 50, the water absorption decreases substantially. Hydration of cement grade 50 produces a higher amount of CSH gel, making the concrete more compacted, leading to fewer pores and voids, making the concrete less likely to absorb water.

The water absorption of concrete must be no more than ten percent, as per BS EN 1992-1-1:2004. At both 7 and 28 days, the water absorption of all five mixture proportions is less than 10%. This suggests that the standard has been satisfied. 15SI-4ASA-10GP-0.3STF-S1.18-G50, the optimal mix, successfully satisfied UHPC specifications by achieving a water absorption of 1.19% at 28 days. Choi et al. (2023) successfully produced Ultra High-Performance Concrete using materials like cement, fly ash, silica fume, quartz powder, recycled sand, water, superplasticiser, and steel fibre, which achieve compressive strengths ranging from 100 MPa to 128 MPa. They performed water absorption tests as well to evaluate their UHPC 's durability. Their water absorption test shows that their mix ranges from 2% to 3%. This indicates that the mix 15SI-4ASA-10GP-0.3STF-S1.18-G50 successfully meets UHPC's requirements.

4.4.5 Density Test

The density of concrete is another important property. High density ensures that there are fewer voids in the concrete, resulting in high strength. The densities of 5 different cementitious composites at 7 days and 28 days are shown in Figure below.



Figure 4.7: Density of Different Cementitious Composites in Phase 3.

The first mix, 15SI-0.3STF-S1.18-G30, serves as a control. It achieves a density of 2280 kg/m³ and 2360 kg/m³ at 7 days and 28 days, respectively. The second mix (15SI-10GP-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 10 percent of the plasticising accelerator with glycerine pitch. Both the density at 7 days and 28 days exhibit a slight increase. The workability and ease of compacting of the concrete are enhanced when 10% of the plasticising accelerator is replaced with glycerine pitch. This results in a more compacted concrete with fewer voids, which in turn leads to a higher density.

The third mix (15SI-4ASA-0.3STF-S1.18-G30), as compared to the first mix (15SI-1STF-S1.18-G30), replaces 4 percent of the sand with alum sludge ash. Both the density at 7 days and 28 days exhibit a slight increase. Due to its high fineness, alum sludge ash can achieve a void-filling effect, thereby reducing the presence of

voids and resulting in denser concrete. This finding is also consistent with that of Kaish et al. (2021), who found that treated alum sludge has the ability to increase concrete's compressive strength when 0 to 10% of the fine aggregate is replaced by treated alum sludge.

The fourth mix (15SI-4ASA-10GP-0.3STF-S1.18-G30), as compared to the first mix (15SI-0.3STF-S1.18-G30), replaces 4 percent of the sand with alum sludge ash and 10 percent of the plasticising accelerator with glycerine pitch. Both the density at 7 days and 28 days also exhibit a slight increase. The combined effect of glycerine pitch and alum sludge ash further increases the density of concrete.

The fifth mix (15SI-4ASA-10GP-0.3STF-S1.18-G50), as compared to the fourth mix (15SI-4ASA-10GP-0.3STF-S1.18-G30), changes from the use of cement grade 30 to cement grade 50. Both the density at 7 days and 28 days exhibit a significant increase. Cement grade 50 features a higher clinker content, leading to greater production of CSH gel, the primary binder in the concrete. This leads to denser microstructure, thus increasing the density of concrete.

According to Sudholt-Wasemann (n.d.), the density of Ultra High-Performance Concrete should range from 2480 kg/m³ to 2790 kg/m³. The fifth mix (15SI-4ASA-10GP-0.3STF-S1.18-G50), which is the optimal mix among these five, achieves a density of 2520 kg/m³ at 28 days and meets the requirements of UHPC.

4.4.6 A Comparison to the Specification of Ultra High-Performance Concrete

Throughout the 3 phases of initial mix development, optimisation, and validation, the optimal mix obtained in this mix is 15SI-4ASA-10GP-0.3STF-S1.18-G50. Various properties of this mix were compared to the specification of UHPC (as shown in Table below). This mix meets the UHPC 's standard in terms of flexural strength, water absorption, and density. However, it falls short in compressive strength and workability.

One interesting finding is that this mix meets the UHPC standard in terms of flexural strength but falls short in terms of compressive strength. It is mainly because of the presence of steel fibre. Steel fibre works by bridging the cracks, distributing the stress across the materials, and inhibiting the microcracks from becoming macrocracks. Steel fibre is primarily used to enhance the tensile and flexural strength of materials; however, it can also enhance compressive strength, although its effect on compressive strength is less significant than that of tensile and flexural strength.

This theory is confirmed by the research conducted by Song and Hwang (2004), which demonstrates the use of steel fibre to create high-strength concrete. Based on the volume fraction, Song and Hwang (2004) incorporate steel fibre into concrete at an amount of 0.5%, 1.0%, 1.5%, and 2.0%. Their results suggest that the compressive strength improves by 7.1% at a 0.5% fraction, 11.8% at a 1.0% fraction, 15.3% at a 1.5% fraction, and reduces to 12.9% at a 2.0% fraction. The flexural strength improves by 28.1% at a 0.5% fraction, 57.8% at a 1.0% fraction, 92.2% at a 1.5% fraction, and 126.6% at a 2.0% fraction. Their findings suggested that steel fibres contributed significantly to the increase in flexural strength, but they had little effect on the development of compressive strength.

	Optimal Mix in	UHPC'	Meet the
	This Study	requirement	requirements?
Flow Table, mm	156	>200	No
Compressive Strength, MPa	77.781	>120	No
Flexural Strength, MPa	16.01	>15	Yes
Density, kg/m ³	2520	>2480	Yes
Water Absorption, %	1.19	<3	Yes

 Table 4.1: Specification of Ultra High-Performance Concrete.

4.5 Scanning Electron Microscopy

Scanning electron microscopy (SEM) was utilised to evaluate the change in the microstructure of the samples as alum sludge ash or glycerine was added, as well as when cement grade 30 changed to grade 50. All the samples developed in phase 3 were studied for their microstructure. Samples for SEM analysis were acquired by striking concrete cubes and collecting their small fragments. As a result, the surface of all samples is shown to be irregular in the images. The evaluation was done by visual observation of the images generated by the SEM equipment. All the images generated for 5 samples are shown in Figure 4.8 below.

The 15SI-0.3STF-S1.18-G30, which lacks alum sludge ash and glycerine pitch, appears to have voids and pores present. Glycerine pitch replacement at 10% (15SI-10GP-0.3STF-S1.18-G30) has shown improvement of microstructure as compared to (15SI-0.3STF-S1.18-G30). This serves as evidence that glycerine pitch improves workability and makes concrete more compact with fewer pores.

Also, compared to control samples (15SI-0.3STF-S1.18-G30), alum sludge ash replacement at 4% (15SI-4ASA-0.3STF-S1.18-G30) shows slightly fewer pores and voids. This suggests that alum sludge ash has the ability to fill voids, slightly reducing the number of pores and voids.

The addition of both glycerine pitch and alum sludge to the mix further reduces the number of pores and voids in the concrete, as indicated in the image of 15SI-4ASA-10GP-0.3STF-S1.18-G30.

When transitioning from cement grade 30 to grade 50, the final mix, 15SI-4ASA-10GP-0.3STF-S1.18-G50, demonstrates the least number of pores and voids and the smoothest microstructure. It is evident that the microstructure is substantially enhanced by upgrading the cement grade. This microstructure is desirable in concrete because it ensures concrete with greater durability and strength due to the reduced number of pores and cavities.





(SEM) Analysis.

4.6 Life Cycle Assessment

Whenever a product is developed, it always inevitably comes with some environmental adverse impact, like gas emissions, waste generation, or natural resource exploitation. If these environmental impacts are not addressed, ongoing development will worsen the impact on a larger scale and to a greater extent, resulting in environmental degradation. Concrete development is no exception. Plus, ultra-high-performance concrete uses even more raw materials than traditional concrete, leaving a higher extent of environmental impact. Therefore, it highlights the pressing need to develop a green UHPFRCC. By using an evaluating tool like Life Cycle Assessment to quantify the environmental impact associated with the production of UHPFRCC, it helps to develop a greener UHPFRCC.

4.6.1 Embodied Carbon (EC) and Embodied Energy (EE)

Embodied carbon and embodied energy are the parameters used to quantify the environmental impact that occurs throughout the life cycle of a product or system, which involves the extraction of raw materials, transportation, installation, maintenance, and end-of-life. The amount of carbon emitted during the product life cycle is measured by embodied carbon, while the amount of energy consumed during the product life cycle is measured by embodied energy. Measurement and calculation of these two parameters are critical to assessing the environmental impact of a product's development. It helps to provide an opportunity for researchers to develop a product with less environmental impact, thereby preserving the environment and achieving sustainability. Also, numerous countries in the world have implemented policies that are designed to decrease the carbon emissions and energy consumption associated with construction. Consequently, it is imperative to quantify and report the embodied carbon and energy.

The calculation of embodied carbon and embodied energy is performed based on the optimal mix, which is the fifth mix from phase 3, namely 15SI-4ASA-10GP-0.3STF-S1.18-G50. In this mix, silica fume replaces 15% of the grade 50 cement, alum sludge ash replaces 4% of the sand, glycerine pitch replaces 10% of the plasticising accelerator, and 0.3% of the steel fibre dosage is based on the volume fraction of the concrete.

For glycerine pitch and alum sludge ash, it is assumed that the embodied carbon is zero. As indicated in the literature review, glycerine pitch is typically disposed of at an incineration facility or landfill, while alum sludge is disposed of at the landfill. The incineration of glycerine pitch is notably responsible for the release of a significant amount of carbon dioxide. Landfill disposal further exacerbates environmental degradation by releasing methane, which has a greater warming impact than carbon dioxide, and consuming a significant amount of land. This exacerbates the scarcity issue in Malaysia's landfill, which is rapidly approaching its capacity. Nevertheless, the carbon emissions that are associated with this traditional disposal method (incineration and landfill) are avoided if the glycerine pitch and alum sludge can be reused in concrete production. For these reasons, it is reasonable to assume that the glycerin pitch and alum sludge have zero emissions when reused in concrete production. Despite the fact that the treatment of alum sludge to produce alum sludge ash consumes a lot of energy, the amount of emission is still minimal when compared to the traditional disposal method of alum sludge, which is landfilling.

The table below displays the calculation. As indicated in the table, the total embodied carbon of the optimal mix developed in this study is 0.82 tonnes CO₂ per cubic metre of material. Wang et al. (2024) state that the production of UHPC is associated with high amounts of CO₂ emissions, ranging from 0.68 to 0.85 tonnes per cubic metre of material. As a result, the optimal mix developed in this study met the criteria defined in the study (Wang et al., 2024).

The optimal mix developed in this study has a total embodied energy of 2.505 MJ/kg. Murthy and Iyer's (2014) study aimed to assess the embodied energy associated with ultra-high-performance concrete. They checked the embodied carbon of UHPC mixtures that met the UHPC requirements by changing the amount of cement that was replaced with silica fume, fly ash, and ground granulated blast furnace slag. The findings indicate that the UHPC mixes have embodied energy ranging from 4.167 MJ/kg to 4.2896 MJ/kg (Murthy and Iyer, 2014). This indicates that the optimal mix developed in this study met the requirements.

In terms of both embodied carbon and embodied energy, the optimal mix met the requirement as compared to the standard of ultra-high-performance concrete.

Materials	Quantity to	Embodied carbon	Total Emission,
	produce 1000kg	(kg CO2.eq/kg)	(kgCO ₂)
	of UHPFRCC		
	(kg)		
Cement	373.89	0.8300*	310.3287
Silica Fume	65.98	0.0140*	0.9237
Sand	387.08	0.0025*	0.9677
Alum Sludge Ash	16.13	-	0
Water	139.29kg	0.0003*	0.0418
	(0.13926m ³)		
Plasticizing	7.13	0.7200*	5.1336
Accelerator			
Glycerine Pitch	0.79	-	0
Steel Fiber	9.71	0.8500**	8.2535
Total Carbon Emission, kgCO ₂			325.6490
Embodied carbon, kgCO ₂ /kg			0.3256
Embodied Carbon, kgCO ₂ /m ³			820.6
(Takir	ng density of 2520 k	(m^3)	

Table 4.2: Embodied Carbon of 15SI-4ASA-10GP-0.3STF-S1.18-G50.

* Embodied carbon of materials was obtained from (Adesina, 2020).

**Embodied carbon of material was obtained from (British Tunnelling Society, 2022).

Materials	Quantity to	Embodied energy	Total Embodied
	produce 1000kg	(MJ/kg)	Energy
	of UHPFRCC		(MJ)
	(kg)		
Cement	373.89	5.500	2056.395
Silica Fume	65.98	0.036	2.375
Sand	387.08	0.080	30.966
Alum Sludge Ash	16.13	-	0
Water	139.29kg	0.010	1.393
	(0.13926m ³)		
Plasticizing	7.13	9.000	64.170
Accelerator			
Glycerine Pitch	0.79	-	0
Steel Fiber	9.71	36.000	349.56
Total Emission per 1000kg of UHPFRCC, MJ			2504.859
Embodied Energy, MJ/kg			2.505

Table 4.3: Embodied Energy of 15SI-4ASA-10GP-0.3STF-S1.18-G50.

* Embodied energy of each material was obtained from (Murthy and Iyer, 2014).
4.6.2 Economic Appraisal

The development of ultra-high-performance fibre-reinforced cementitious composites (UHPFRCC) always comes with a very high cost. This is the main obstacle preventing UHPFRCC from being widely used. As such, it necessitates that the costs be calculated.

The cost analysis is performed based on the optimal mix from phase 3 (15SI-4ASA-10GP-0.3STF-S1.18-G50). All the materials are based on the current market price. Only the cost of materials is considered, without considering the costs associated with mixing, transportation, compacting, formwork, and other related operations. Glycerine pitch is a waste, plus it does not require any pre-processing, so it is considered zero cost. Alum sludge is a waste, but it requires calcination under 800 degrees Celsius to turn it into ash. Such a large amount of electricity for preprocessing should not be neglected. The price rate of electricity is based on TnB, which is the nation's primary electricity generation enterprise for industrial pricing. For the furnace to operate for 1 hour, it requires an electricity fee of RM3.96. The furnace can produce 5 kg of treated alum sludge at one time, making the material cost for alum sludge ash.

The cost of production of UHPFRCC is shown in the table below. To produce 1 m³ of UHPFRCC with alum sludge ash and glycerine pitch excluded, it requires RM2016.00. To produce 1 m³ of UHPFRCC with alum sludge ash and glycerine pitch included, it requires RM2041.20. Despite the fact that waste replaces sand and plasticising accelerator, the cost is even higher as a result of the pre-processing of alum sludge. However, the cost increase is negligible, at only 1.25%. According to Wang (2021), the materials for UHPC with fibre reinforcement cost about RM4863.00/m³. This suggests that the UHPFRCC that was developed in this investigation is cost-effective.

Table 4.4: Cost for UHPC Production.

Material	Cost, RM	Quantity used to	Quantity used to	Total Cost, RM	Total Cost, RM
		produce 1000kg of	produce 1000kg of	(Control)	(+ASA&GP)
		UHPFRCC, kg	UHPFRCC, kg		
		(Control)	(+ASA &GP)		
Cement	RM0.45/kg	373.89	373.89	168.25	168.25
Silica	RM6.54/kg	65.98	65.98	431.51	431.51
Fume					
Sand	RM0.04/kg	403.21	387.08	16.13	15.48
Alum	$12 \text{kw} \times 1 \text{h} \times \frac{\text{RM0.33}}{1 \text{ km}} = \text{RM3.96}$	0	16.13	0	12.74
Sludge Ash	RM3.96				
Water	RM2.28/m ³	139.26kg	139.29kg	0.32	0.32
		(0.13926m ³)	(0.13926m ³)		

Plasticising	RM4.04/kg	7.92	7.13	32.00	28.81
Accelerator					
Glycerine	RM0/kg	0	0.79	0	0
Pitch					
Steel Fiber	RM15.75/kg	9.71	9.71	152.93	152.93
	Total Cost for 1000 kg of U	801.14	810.04		
	Cost for 1 kg of UHPFR	0.80	0.81		
	Cost for 1m ³ of UHPF	2016.00	2041.2		
	(Taking density				
	Percentage of inc	1.25			

4.7 Summary

The results from phases one, two, and three of the experiment have been analysed and interpreted. In phase 1 experimental work (first mix development), it is discovered that steel fibre and silica fume contribute to the increase in compressive strength. Glycerine pitch can only partially replace the plasticising accelerator; it cannot replace it completely. Glycerine pitch caused an early strength decrease, but because it made the concrete more workable and compacted, it was able to improve long-term strength. In order to increase the pozzolanic activity of alum sludge, it was necessary to treat it prior to its addition to the manufacturing of concrete. In addition, in terms of long-term strength, sand size (<1.18 mm) is better than sand size (<4.75 mm). The smaller the size of the sand grains, the more corrosion-resistant the steel fibre within.

In phase 2 experimental work (optimisation of mix proportions), it aims to optimise the concrete mix by utilising three factors, each with a different level. Key findings show that an optimal mix consisted of 15% silica fume, 4% alum sludge ash, and 10% glycerine pitch.

In phase 3 experimental work (comprehensive testing and validation), the findings show that concrete performs better in terms of compressive strength, flexural strength, water absorption, and density when 4% sand is replaced with alum sludge ash and 10% plasticising accelerator is replaced with glycerine pitch. Furthermore, alum sludge ash's capacity to absorb water causes concrete to lose workability, but the addition of glycerine pitch increases it. Through three phases of experimental work, the optimal mix that balances between performance, environmental friendliness, and cost is 15SI-4ASA-10GP-0.3STF-S1.18-G50. In terms of performance, this mix meets Ultra High-Performance Concrete's requirements for properties such as flexural strength, water absorption, and density. According to life cycle assessments performed, this mix meets the requirement of UHPC in terms of embodied carbon and embodied energy. The cost of production was also assessed through economic appraisal. RM2041.20/m3 is the result of this mixture. In comparison to the cost of UHPC production, it is highly cost-effective.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

The first objective in this study, which is to investigate the feasibility of using waste materials, namely, glycerine pitch and alum sludge, in the production of green cementitious composites, is achieved. It has been proven that glycerine pitch can partially replace plasticising accelerator, improving the properties of concrete. The plasticising accelerator releases trapped water between granules, and the glycerine pitch acts as a lubricant. When combined, they improve workability and fluidity, which in turn enhance the strength, density, and water absorption of the concrete. It was also proved that alum sludge ash can partially replace sand. This is due to alum sludge ash's internal void-filling effect and pozzolanic activity, which increase concrete strength, water absorption, and density. However, alum sludge ash tends to absorb water, reducing workability and increasing the difficulty of compacting. The workability loss caused by alum sludge ash can be compensated for by the oily characteristic of glycerine pitch when used in conjunction with alum sludge ash in the concrete mix. As a result, the concrete's various properties are further enhanced. These waste materials demonstrated advantageous effects on the properties of concrete, thereby reducing the consumption of raw materials and resolving the waste management issue, thereby accomplishing an environmentally friendly purpose. Consequently, the feasibility of utilising waste materials such as glycerine pitch and alum sludge ash in the production of green cementitious composites has been demonstrated.

The second objective of this study, which is to develop a cementitious composite that is cost-effective, environmentally friendly, and of ultra-high performance, has been mostly accomplished. In this study, the optimal mix was determined to be 15SI-4ASA-10GP-0.3STF-S1.18-G50. It contains a water-to-binder ratio of 0.32, 15% cement replacement with silica fume, 4% sand replacement with alum sludge ash, 10% plasticising accelerator replacement with glycerine pitch, 0.3% steel fibre dosage (based on concrete volume), sand size (<1.18 mm), and cement grade 50. For performance, this mix meets the Ultra High-Performance Concrete's requirements in terms of flexural strength, water absorption, and density. However, it falls short in terms of workability and compressive strength. This mix only partly achieves the standard of UHPC's requirement. Nevertheless, the compressive strength still met the criteria of high-performance concrete, indicating that it possesses strong load-bearing capacity, though not to the level of ultra-high performance. In terms of environmentally friendliness, the optimal mix met the requirement of the UHPC specification in terms of both embodied carbon and embodied energy. In terms of cost, UHPC costs approximately RM4863.00/m³. The optimal mix developed in this study costs approximately RM2041.20/m³. This suggests that this mix is highly costeffective. With all these factors considered, the second objective is mostly achieved. The study successfully demonstrated that the utilisation of waste in the production of high-performance concrete that is both cost-effective and environmentally friendly is feasible.

The study successfully supports certain Sustainable Development Goals (SDGs). The production of high-performance concrete that is both cost-effective and environmentally friendly is in accordance with SDG 9, which is to establish sustainable, resilient, and high-quality infrastructure. Additionally, this study supports SDG 12, which emphasises the significance of sustainable consumption and production patterns. The practice of reusing materials is promoted by the use of such waste in concrete production, which is a key principle of the circular economy. This minimises waste generation. As a result, it mitigates the environmental degradation that results from the overexploitation of natural resources.

5.2 **Recommendations**

To improve the research, several recommendations can be made.

- 1. Reduce the water-to-binder ratio to less than 0.25 while using advanced mixing and compacting equipment and superplasticizer to further improve the compressive strength of UHPFRCC.
- 2. Find the optimal level of steel fibre dosage that balances concrete's performance, cost, and environmental friendliness.
- 3. Investigate whether adding alum sludge ash to UHPFRCC can improve its fire resistance.
- 4. Investigate the durability of UHPFRCC in harsh environments like acidic or alkaline environments, aggressive chemicals, and freeze-thaw cycles.

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