CORRELATION OF SUSTAINABLE MATERIAL IN CONCRETE WITH COMPRESSIVE STRENGTH OF GRADE 25 WITH METAKAOLIN

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A project report submitted in partial fulfilment of the requirements for the award of Bachelor of Civil Engineering with Honours

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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

I certify that this project report entitled **"CORRELATION OF SUSTAINABLE MATERIAL IN CONCRETE WITH COMPRESSIVE STRENGTH 25MPA WITH METAKAOLIN"** was prepared by **TEO CHUNG JING** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Civil Engineering with Honours at Universiti Tunku Abdul Rahman.

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ABSTRACT

Rapid urbanization and population growth have led to a surge in demand for concrete structures and exacerbating environmental issues such as wastage pollution and greenhouse gas emissions. Plastic wastes were the most serious caused of the environmental impact. Hence, this study aims to investigate the usage of sustainable waste materials and pozzolanic materials in the concrete design to achieve the sustainable development goal. Polyethylene Terephthalate (PET) and Metakaolin (MK) was adopted in concrete mix design as PET was the most produced waste plastic among all the plastic types. The effect and incorporation of PET flakes replacing coarse aggregates and MK replacing OPC in concrete mix was determined in terms of fresh properties and hardened properties of concrete, such as slump test, density determination, compressive and splitting tensile strength test. Firstly, during trial mix, both CTR and PET-5 with different W/C ratio was prepared and the concrete mix with W/C ratio of 0.6 was selected to be optimal concrete mix design for further experimental work as both CTR and PET-5 had achieved the desired strength of 25 MPa. In Phase 2, three design concrete mixes were prepared with different percentages of PET replacement from range 5 % to 15 % with interval of 5 %. Thus, PET-10 was selected as optimal concrete mix design and adopted in Phase 3. In Phase 3, PET-10 was mixed with different percentages of MK replacement from 5 % to 10 % with interval of 2.5 %. PET-10 +MK-7.5 exhibited the greatest compressive strength of 37.71 MPa by increased 27 % of PET-10 with compressive strength of 29.71 MPa due to the pozzolanic effect of MK that can increase the concrete strength. Furthermore, the microstructural analysis, such as SEM-EDX and XRD analysis, were performed to investigate the chemical composition of the designed concrete. Consequently, the PET-10 + MK-7.5 was the optimal design concrete mix to achieve the sustainability goal and concrete strength of 25 MPa.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS / ABBREVIATIONS

Ac	cross-sectional area of cubic specimen, m^2
B	average width of specimen, mm
D	diameter, mm
fc	compressive strength, MPa
F	peak load, N
L	specimen’s length, mm
P	pressure, kPa
T	splitting tensile strength, MPa

ASTM	American Society for Testing and Materials
BS EN	British Europe
CTR	Control Concrete
EDX	Energy Dispersive X-ray
MK	Metakaolin
OPC	Ordinary Portland Cement
PET	Polyethylene Terephthalate
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 General Introduction

Concrete is widely used in the construction industry due to its unique combination of workability, durability, strength and the availability of raw materials. Typically, the concrete consists of cement paste, water, and aggregates in varying proportions. The process of concrete mix proportioning, also known as mix design, involves selecting suitable ingredients, determining the water-cement ratio, and adjusting the quantities to achieve the desired workability, strength, durability, and other specific requirements in accordance with ASTM standards. This process can be more complex by incorporating additional ingredients with different properties, such as admixtures, sustainable materials, on-site solid waste and more, in order to meet specific design criteria (Rosa et al., 2023).

Recently, rapid urbanization and population growth around the world have led to an increase demand for new infrastructure and housing that required enormous amounts of raw materials of concrete structure (Nilimaa, 2023) and the increasing demand of landfill for waste disposal to its essential asset of human consumption such as plastic consumption. From an environmental perspective, concrete cement manufacturing and casting have a significant impact on the greenhouse effect and the increased demand for plastic consumption lead to plastic pollution, landfills, or marine debris. In Malaysia, the emergence of marine pollution and landfills have become a genuine issue that treatment is needed urgently. These problems are due to the rapid economic growth in Malaysia, causing an increase of 4% waste generation annually. Plastic waste is the main cause to these environmental pollutions with an average plastic consumption of 56 kilograms per capita per year. Malaysia has ranked 20th among the world's top plastic polluters in 2021 (Fauziah et al., 2021).

Therefore, pozzolanic materials such as fly ash (FA), ground granulated blast furnace slag (GGBS), or metakaolin (MK), can be a potential substitute for Ordinary Portland Cement (OPC) to develop a geopolymer concrete. These materials are rich in silicon (Si) and aluminium (Al) that are beneficial to the mechanism strength of concrete. Utilization of metakaolin in industry is considerable for partial replacement of OPC powder to achieve an energy-saving, reduce greenhouse gases effect and enhance the performance of cementitious composites. There is a substantial 43% to 59% reduction in energy consumption during production compared to traditional concrete (Albidah et al., 2021).

On the other hand, daily human activities generate a large volume of plastic waste, encompassing plastic types like Polyvinyl Chloride (PVC), Polypropylene (PP), Polyethylene Terephthalate (PET) and more. The plastic types of Polyethylene Terephthalate (PET) and High-Density Polyethylene (HDPE) are adopted as substitutes for aggregate or as fibres in concrete aiming to reduce the adverse environmental effects of plastic waste. This integration of PET into the concrete mix offers a cost-effective approach to sustainable concrete and alters the properties of cement concrete such as exhibiting rapid curing and workability (Waysal et al., 2022).

To further observe on the material properties of the mixed concrete in detail, Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) are utilized to study the effects of microstructure and mechanical properties of concrete. SEM is an effective analytical method capable of examining a wide range of materials at high magnifications, allowing for the capture of high-resolution images. While EDX analysis stands out as one of the most dependable methods. These techniques offer a reliable qualitative and quantitative evaluation of the hydrated products, making products used in this study (Singh et al., 2023). Moreover, X-ray Diffraction (XRD) is also used to determine the atomic and molecular compound.

1.2 Importance of the Study

In recent years, the issues of global warming and environmental pollution in terms of landfills and marine pollution have emerged as crucial topics requiring immediate attention worldwide. According to Ansari et al. (2023), the manufacturing of ordinary Portland cement concrete (OPC) is responsible for 30% of the global CO_2 emissions from industrial and energy sources. Meanwhile, the Environmental Protection Agency reported that out of millions of metric tonnes of plastic waste produced each year, only a mere 7% to 8%

undergo recycling or incineration, with the majority being sent to landfills (Jawaid et al., 2023).

Therefore, the utilization of sustainable construction materials has gained significant attention in recent years due to growing environmental concerns and the need for more eco-friendly practices in the construction industry. In the civil engineering field, researchers have actively sought solutions by innovatively utilizing sustainable materials as a partially replacement of ordinary cement concrete or natural fine and coarse aggregates in construction. For instead, the plastic bottle village located in the island of bocas del toro, Panama by Robert Bezeau. Millions of plastic bottles wee collected and used to build a serios of structure.

However, appropriate proportions of raw materials in concrete mix pose a challenge to achieve the same concrete grade as traditional concrete mix. The potential of this study lies in its ability to propose an optimum combination of PET and metakaolin for a sustainable concrete mix design that can achieve the desired mechanical strength. Successfully formulating this eco-friendly concrete mix design will help reduce dependence on OPC and natural fine and coarse aggregates, effectively tackling the problem of climate change caused by global warming. Additionally, it will create a cost-effective scenario that will attract stakeholders for commercialization purposes.

1.3 Problem Statement

The construction industry in Malaysia is currently confronted with the challenge of reducing the environmental impact while maintaining the required structural performance of concrete. However, construction sectors are rarely considered to adopt sustainable concrete on-site due to the complexity and expense of searching to replace traditional cement. In response, researchers and industry professionals are continuously exploring suitable sustainable materials to substitute conventional construction materials to solve the concern of the construction sector.

First and foremost, it is crucial to study the compatibility between the chosen waste PET, metakaolin and conventional cementitious matrix on fresh and hardened properties of concrete to ensure that the sustainable concrete mix meets the desired strength, workability and durability. By far, there are insufficient research articles on the related objective for obtaining the results and relevant references.

Furthermore, the second problem is extensive experimental and analysis are required to establish the optimal mix proportions of PET as coarse aggregate replacement and metakaolin as a partial cement replacement to achieve the desired compressive strength. However, the availability of research on related sustainable concrete mix design is limited, due to the recent global emphasis on sustainable development and the relatively new emergence of the concept of creating sustainable concrete structures.

Lastly, OPC and concrete mixed with PET and metakaolin theoretically showed the differences in the mechanical properties among them. For instance, mechanical strength, durability and workability. Nevertheless, it may be interesting to study and compare the mechanical properties of these two different properties of concretes while maintaining the achievement of required compressive strength.

1.4 Aim and Objectives

The aim of the study is to innovate and obtain the mix proportion for sustainable material such as PET to replace the coarse aggregates and metakaolin with the replacement of OPC to obtain the desired compressive strength. The objectives are shown as follows:

i. To design the mix proportion with concrete grade 25MPa.
ii. To investigate the optimal percentage between 5 %, 10 % and 15 % replacement of PET for coarse aggregate and effect to achieve concrete grade 25 MPa compressive strength.
iii. To synthesize the effect of metakaolin replacement for OPC under 5 %, 7.5 % and 10 %.

1.5 Scope and Limitation of the Study

The scope of this study was to propose an optimal mix proportion for concrete mix and study the concrete properties and correlation of the proposed concrete mix. Firstly, it is required numerous experiments to obtain reliable results of the optimal concrete mix proportion for PET flakes as sustainable materials and metakaolin as partial replacement of OPC to achieve the desired compression strength of 25MPa. The “Orang Kuat” Type I cement (OPC) was used in concrete mix as it was easy to obtain and has been certified with ASTM standards.

After the concrete mix design has done, a study on the concrete properties of the proposed concrete mix was carried out. The study primarily examines the fresh and hardened properties of sustainable concrete including the slump test for fresh properties test, and compression test and splitting tensile test for hardened properties test. The microstructures of the sustainable concrete are also to be analysed by using the methods of SEM, EDX and XRD. The tests and analysis were performed to investigate the correlation between PET and MK to achieve desired compressive strength of 25 MPa.

The limitation of the study is the challenge of achieving identical results using the same mix design with PET and from different manufacturers. They produced by various companies may exhibit significant differences in micromechanical properties, posing a potential risk to the overall outcomes of the study. Furthermore, PET was limited to partial replacement of the coarse aggregates in this study as the size of the PET obtained were sized within the range of 5 mm to 15 mm.

1.6 Contribution of the Study

This research provides the framework for future research and developments in optimizing the concrete mix using PET as sustainable materials and metakaolin as a replacement for natural coarse aggregates and OPC. The developed concrete mix design of PET and metakaolin improves the workability of cement during the fresh concrete phase, the strength and durability of concrete after hardening to achieve the desired compressive strength of 25 MPa. The usage of PET and metakaolin in concrete mix can change the properties of the concrete compared to the conventional OPC without any replacement.

PET as the sustainable materials used in concrete mix design. Reuse of plastic waste of PET in the concrete construction sector to reduce landfill disposal and marine debris. This contributes to the achievement of sustainable development goal 11, which aims to create sustainable cities and communities. The metakaolin is the replacement for cement to increase the compressive strength of concrete and reduce the environmental impact such as the greenhouse effect due to CO_2 emission during the construction and manufacture of OPC.

This study has discussed the factors affecting optimal mix concrete proportion and properties of fresh and hardened concrete. Which is the percentage of PET used to replace for coarse aggregates and metakaolin to replace for OPC. To investigate the properties of the concrete mix and correlation of the PET and metakaolin. The method of SEM, EDX and XRD were used to study the microstructure of the concrete mix and the universal compressive machine was suitable to conduct the destructive test. Thus, this study provides the opportunity to reduce environmental pollution and construction field able to reuse plastic waste and metakaolin as replacements for coarse aggregate and to develop an sustainable concrete design.

1.7 Outline of the Report

This final report is divided into five chapters: introduction, literature review, methodology, results and discussions, and conclusion and recommendations.

In Chapter 1, a general introduction on concrete mix design with sustainable materials and the metakaolin, and the importance of the study is provided. The problem statement, aim and objective, scope and limitation of the study are determined and discussed.

In Chapter 2, discussed the detail literature review on environmental pollution regarding to plastic waste and CO_2 emission, fresh properties and hardened properties of concrete that are mixed with PET coarse aggregates as sustainable material, metakaolin as pozzolanic material as substitution materials of OPC. SEM, EDX and XRD analyses on the surface and elemental composition of concrete is literature reviewed.

In Chapter 3, the procedures of raw materials preparation and concrete mixing were described in detail and the mixing proportion with admixture of PET and metakaolin were listed. This study also included the slump test method for fresh properties test. While, the compression test and splitting tensile test were conducted for the hardened properties test. SEM, EDX and EDX analyses on the surface and elemental composition of concrete are carried out. The tests are performed in accordance with ASTM standards.

In Chapter 4, after a series of trial mixes are done. The real mixes were then conducted. The results of fresh properties test, hardened properties test and microstructure analysis were recorded and discussed the optimum proportion of PET and metakaolin that used for the replacement of coarse aggregates and OPC to achieve the compressive strength of 25 MPa.

Lastly, in Chapter 5 presents the conclusion of the project and recommendations for future study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter provides a general review, starting with an introduction to environmental impacts due to human activities across the world, through urbanization, mining, exploration and industrialization. According to this study, it is mainly focused on the environmental pollution caused by concrete manufacturing, construction and wastage. To reduce the adverse impact of concrete, metakaolin as a geopolymer has been chosen as a replacement for OPC and possible sustainable materials that can be reused in concrete casting such as plastic, food waste, and rubber.

As commonly recognized, plastic waste is one of the most troublesome forms of waste that requires treatment. As a result, plastic waste has been chosen as a sustainable material to be integrated into concrete casting in order to improve environmental conditions. The utilization of plastic in concrete casting provides several advantages, including greater workability. Therefore, the types of plastic waste that has been chosen in concrete casting are PET and HDPE as their wide availability, allowing them easily to obtained from various recycling centre.

Next, according to the ASTM standard, the concrete mix proportion is important as the standard given the guideline of concrete mix included the ratio of w/c, fine aggregates, coarse aggregate and OPC to create a conventional concrete that can provide certain strength for construction structure. However, the innovative concrete mix with the replacement of PET and metakaolin and has promoted sustainable concrete. However, it is required to meet the requirements such as good workability and maintain the compressive strength of 25 MPa.

Lastly, the microstructure of the concrete mix with various types of materials is studied through SEM, EDX and XRD analyses.

2.2 Environmental Pollution

In Malaysia, there has been a recent problem of environmental pollution due to the growing waste generated from human activities by 4 % per year. Example of this include pollution of the marine environment, poorly managed landfills and air pollution. First and foremost, marine pollution is caused by submerged, sinking and floating debris. Initially, the wastes are disposed of by burying them in a landfill or incineration to ash properly. However, there are several wastes end up in waterways and oceans, particularly plastic wastes due to inadequate waste management (Fauziah et al., 2021). According to Grand View Research, the plastic compounding market in Malaysia is valued at USD 905.2 million in 2022 and is predicted to expand at a compound annual growth rate of 7.6 % from 2023 to 2030 as shown in Figure 2.1.

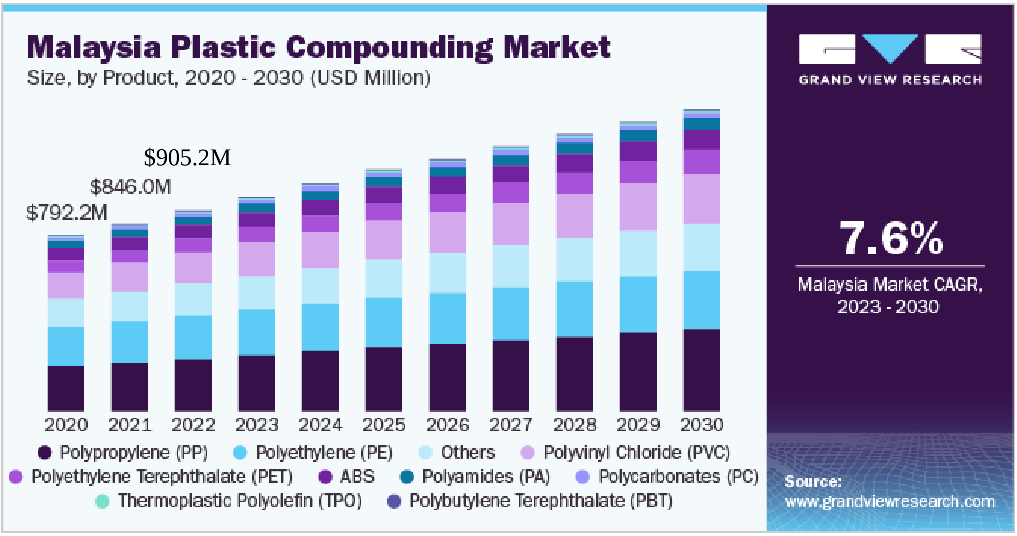


Figure 2.1: Malaysia Plastic Compounding Market Size from 2020 to 2030 (Grand View Research, 2023).

The mismanaged plastic waste in the ocean has broken into <5mm micro size particles and further impacts on marine ecosystems adversely, such as penetrating the marine food chain through the ingestion by organisms. It is highlighted that several marine species have been found are ingested microplastic into the body which leads to rising concern regarding the potential impacts of microplastics on human well-being (Fauziah et al., 2021).

Apart from that, landfilling is one of the prevalent and cost-effective methods for municipal solid waste disposal worldwide. The municipal solid waste includes product packaging, construction waste and bottles. After a period of rainfall and microbial degradation, 10–20% of leachate has been generated from the waste mass under moisture conditions. The leachate contains various pollutants such as inorganic salts, heavy metals and microorganisms that are hazardous to the environment. Poorly managed and inadequate landfilled will cause the leachate infiltrate into the soil and endangering surrounding ecosystems and threatening human health (Sha et al., 2023). Figure 2.2 shows that the seepage of leaked landfill leachate into groundwater has reduced the stability and efficiency of microbial communities and increased the vulnerability of the stable microbial ecosystem.

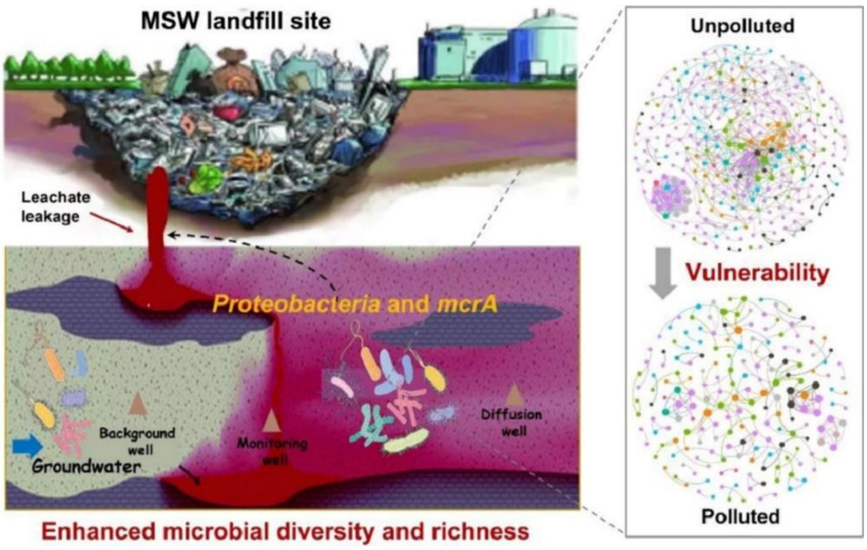


Figure 2.2: Adverse Effect of Leachate Seepage into Groundwater (Sha et al., 2023).

Nevertheless, increasing demand for cement manufacturing is also a major concern of environmental pollution. Figure 2.3 shows that the growing global cement production from 2010 to 2021. Cement manufacturing mainly contributes to the greenhouse effect and air pollution. This is because the production of cement requires a lot of energy to dry, crush, blend and heat the cement raw materials such as limestone and gypsum. Figure 2.4 illustrates the various types of energy sources that are used to generate the electrical power to heat the cement's raw materials up to 1200 - 1450 ºC. Among these energy sources, coal was the main energy source used with 53% (Sahoo et al., 2022), where the burning of coal is one of the major causes of the greenhouse effect and air pollution.

Figure 2.3: World Cement Production from 2010 to 2021 (Sahoo et al., 2022).

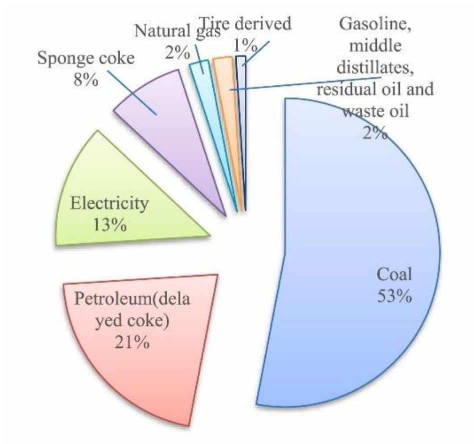


Figure 2.4: Electrical Energy Utilization in Cement Production by Energy Source (Sahoo et al., 2022).

During the combustion of fuels, various types of gases or tiny particles are emitted into the atmosphere such as nitrogen gases (NO_x), carbon dioxide (CO_2) and Sulphur dioxide (SO_2). The cement industry has contributed around 5 % of global CO_2 emissions. Mainly released during the carbonates' calcination in raw materials with 50 to 60 % and fossil fuel combustion with 40 %. Additionally, there is 5 to 10 % of indirect CO_2 emitted from the cement industry such as electricity consumption for powering plant equipment and transportation (Karstensen et al., 2016). Excessive CO_2 emissions attributes the greenhouse effect which leads to climate change.

2.3 Sustainable Materials

Currently, there is a gradually growth in public awareness about environmental protection and resource conservation in Malaysia. For example, solid waste, agricultural waste and food waste can be managed by appropriately monitoring human activities or reusing these wastes as sustainable materials instead of being disposed into the landfill. The use of sustainable materials in construction work not only reduce earth pollution by limiting the waste disposal but also reduces the CO_2 emission of cement during the manufacturing process and construction industry by using sustainable materials as partial replacement of conventional concrete materials. Modern construction work is also focusing on natural resource conservation and sustainable growth to preserve the environment (Jahami and Issa, 2023).

The recycling of plastic waste to partially replace fine or coarse aggregates in conventional concrete mixture has been extensively studied to achieve sustainable development goals. In term of environmental concern, plastic waste is one of the biggest problems, especially ocean pollution. In Figure 2.5 shows all types of plastics for various uses, such as Polyethylene Terephthalate (PET), High-Density Polyethylene (HDPE), Polypropylene (PP) and Polystyrene (PS). Where the PET, HDPE are commonly recycled as there is a strong market for their by-products used to produce water bottles or shampoo bottles (Brown Recycling, 2022).

Figure 2.5: Infographic of 7 Types of Plastics (Brown Recycling, 2022).

2.3.1 Polyethylene Terephthalate (PET)

One of the major components of plastic waste is polyethylene terephthalate (PET). PET is a synthetic polymer commonly used in plastic bottles as shown in Figure 2.6 (Aproko247, 2018). Due to its low cost, ease of handling and lightweight nature, the increased demand for PET has led to a tremendous unmanaged plastic waste that has disposed into the marine which causes serious environmental pollution. To reduce the plastic pollution, the recycling content is an excellent option, however, process of making new products from degraded waste is cost inefficiency. So, the alternative method is required to utilize the plastic waste from deposed into landfill and marine. The idea of utilizing the waste PET bottles as coarse aggregates in concrete is expected to carry out positive effect in term of protection of environmental containment (Dipta and Rahat, 2018). Figure 2.7 shows the fine pieces of PET has blinded and prepared to replace the natural coarse aggregates partially.

Figure 2.6: PET Waste of Water Bottle (Aproko247, 2018).

Figure 2.7: Fine pieces of PET (Dipta and Rahat, 2018).

In the concrete mixture with the partially replacement of PET to coarse aggregates, the fresh concrete properties have studied by (Bamigboye et al., 2022). The study carried out both slump cone and compaction factor test to assess the workability and consistency of the concrete mix in accordance with the standards of ASTM C143/C143M-15a. Based on the Figure 2.8, indicated that the workability increased as the proportion of PET in the mix has increased. All concrete mix exhibited true slumps, except for the 100 % of PET indicated early-age collapse or shearing of concrete. The increase in workability with higher PET content can be attributed to reduce in stiffness as the PET aggregates have lower water absorption capacities. Which can be resulting in weaker binding between the matrix and PET.

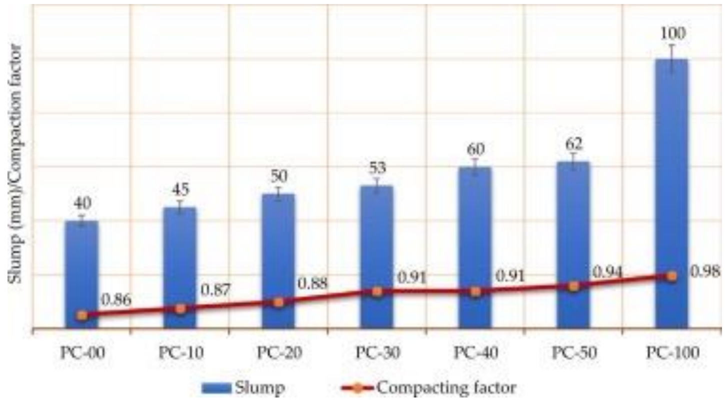


Figure 2.8: Slump and Compacting Factor Test of Fresh Concrete with various PET content (Bamigboye et al., 2022).

However, Table 2.1 shows the research that carried out Saikia and De Brito (2014) exhibited that increases in PET aggregates with fine, coarse or spherical shapes was decreases the slump value compared to the reference results due to the sharp edges and angular particle shape of PET.

Table 2.1: Fresh Concrete Properties of PET with Coarse, Fine and Spherical Shape (Saikia and De Brito, 2014).

Properties	**Ref**	**PC**			**PF**			**PP**		
		5	**10**	**15**	**5**	**10**	**15**	**5**	**10**	**15**
W/C (%)	0.53	0.61	0.65	0.74	0.57	0.60	0.64	0.53	0.52	0.52
Slump (127)	127	120	120	-	122	122	120	122	122	132
Density (kg/m^3)	2387	2326	2277	2233	2336	2290	2243	2347	2297	2254

According to the experiment conducted by Supit and Priyono (2022), the concrete mix with PET coarse aggregates with the sizes of 5-10 mm and 10-20 mm respectively. The coarse aggregate was partially replaced by 5 % of recycled PET aggregates while maintaining the cement content. Based on the Figure 2.9, the results obtained has indicated that the addition of PET aggregates has increased the concrete's early strength development from 8.4 to 9.2 MPa but decreased the concrete's compressive strength from 12.9 MPa to 10.3 MPa for aggregate size of 5-10 mm after 28 days. Meanwhile the results taken for aggregate size of 10-20 mm introduced similar trend. This is explained by the lack of adhesion between the cement matrix and the smooth-surfaced PET aggregates. Also, the hydrophobic nature of PET can hinder cement hydration by restricting the water movement.

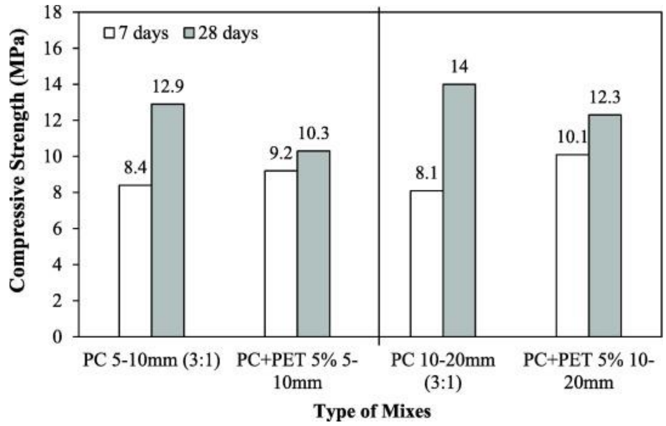


Figure 2.9: Compressive Strength of Concrete Mix with 5 % PET Content (Supit and Priyono, 2022).

The literature reviewed on the research conducted by Khajuria and Sharma (2019), showed the compressive strength, split tensile strength and flexural strength of the C25 grade of concrete with varying proportion of plastic replace the coarse aggregates. M1, M2, M3, M4 and M5 represented the 0%, 2.5 %, 5% and 7.5% of plastic replacement respectively. According to the results obtained, the compressive strength of the concrete decreased as the increased of plastic content for 28 days. Next, the split tensile strength of the concrete has increased with the greater plastic content but was reduced after 2.5% of plastic content. Lastly, concrete's flexural strength also shared the same trend with compressive strength.

2.3.2 High-density Polyethylene (HDPE)

Besides PET plastic wastes, high-density polyethylene (HDPE) plastic wastes are also considered as the popular plastic wastes that has greatly affected the environment negatively such as marine debris. This is because the HDPE raw material is highly demanded in industries that are manufacturing the detergent, pipes and fuel container. The HDPE has the characteristics of light weight, stronger than PET and impact resistant that allowed it to be widely used as packaging bottles and piping materials. Hence, the highly manufactured HDPE without proper disposal management or recycle, it can lead to an environmental pollution, which is similar to PET plastic wastes.

According to the study conducted by Philomina (2017), HDPE has been chosen as the replacement materials for coarse aggregates. Due to easy availability of HDPE, enhance the concrete properties and reduce costs. The usage of plastic waste in concrete introduced the sustainable concrete and contribute to a greener environment. Based on the Table 2.2 showed the values of slump test with various percentages of coarse aggregates replaced by HDPE. The results indicated that higher percentages of HDPE lead to greater slump test values as the HDPE has low water absorption capacities. Meanwhile, the compressive strength of the HDPE shared the similar trend as PET. For instance, Table 2.3 showed the overall decreased in compressive strength of sustainability concrete with various content of HDPE.

Table 2.2: Results of Slump Test for various HDPE Contents (Philomina, 2017).

Mix	**Percentage of HDPE Replaced (%)**	**Slump (mm)**
HDPE1	8	108
HDPE2	16	110
HDPE3	24	115
HDPE4	32	118

Table 2.3: Results Compressive Stress for various HDPE Contents (Philomina, 2017).

Mix	**Percentage of HDPE Replaced (%)**	**Compressive Strength (N/mm^2)**	
		7 days	**28 days**
HDPE1	8	22.50	40.23
HDPE2	16	21.18	37.66
HDPE3	24	16.66	35.54
HDPE4	32	14.84	30.98

Based on the study carried out by Abu-Saleem et al. (2021), the physical properties of the HDPE have obtained with the specific gravity of 0.96 and density of 960.48 kg/m^3. The indirect tensile strength of the concrete with 10%, 20% and 30% of HDPE content were obtained with 2.52 MPa, 2.3 MPa and 2.08 MPa respectively. Which is lesser than the control concrete with 3.26 MPa and gradually decreased with the increased HDPE content. The factor that leads to this phenomenon is the weak bonding strength between the plastic and the cement matrix. Besides, the flexural test also shared the same trend with indirect tensile test. Gradually decrease of flexural strength of concrete with the increased plastic content from 3.54 MPa to 2.99 MPa compared to control concrete with 4.29 MPa.

2.4 Metakaolin

According to ASTM C 618, metakaolin is entirely naturally occurring mineral that contained high SiO (50–55%) and AlO (40-45%), while the other supplementary cementitious materials are the byproduct produced from manufactural industry, such as fly ash and silica fume. The production of metakaolin is required to produce with high-quality standard, ensuring a uniform composition, enhance the pozzolanic reactivity and increase purity compared to other manufactured pozzolan. In the concrete mixing process, the metakaolin is partially replaced the cement and reacts with water content to carried out cement hydrates process, more C-S-H gel is formed, and the strength of the concrete is greatly increased, particularly at early stages. This is known as pozzolanic effect. It is because the metakaolin consisted of fine particles that effectively occupy the gaps between cement practices, resulting in a denser cement matrix. Thus, the densification occurred has reduced the water and oxygen infiltration, enhanced the resistance to chloride ion diffusion, acid and sulphate attacks, and promoted the overall concrete strength and durability (Elhadi et al., 2023). Figure 2.10 shows the increases in compressive strength of concrete with the increases in metakaolin (M) contents. Furthermore, the tensile strength of the concrete is increased with the increased of metakaolin added as shown in Figure 2.11.

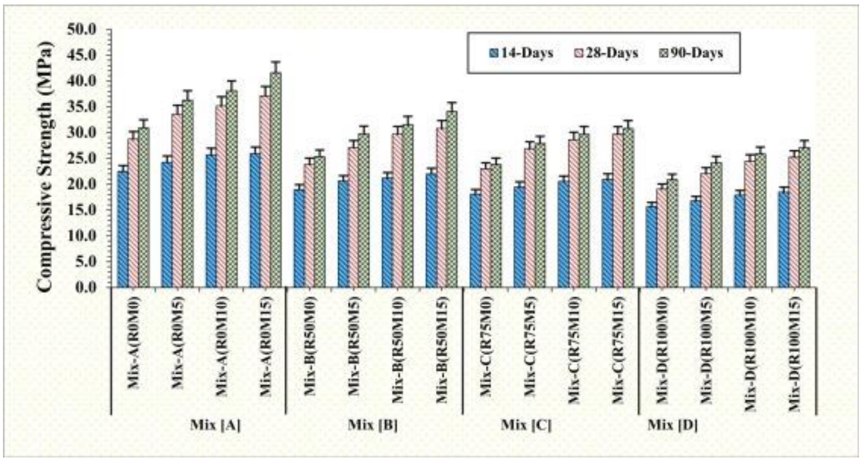


Figure 2.10: The Compressive Strength of Concrete with different Portion of Metakaolin used (Elhadi et al., 2023).

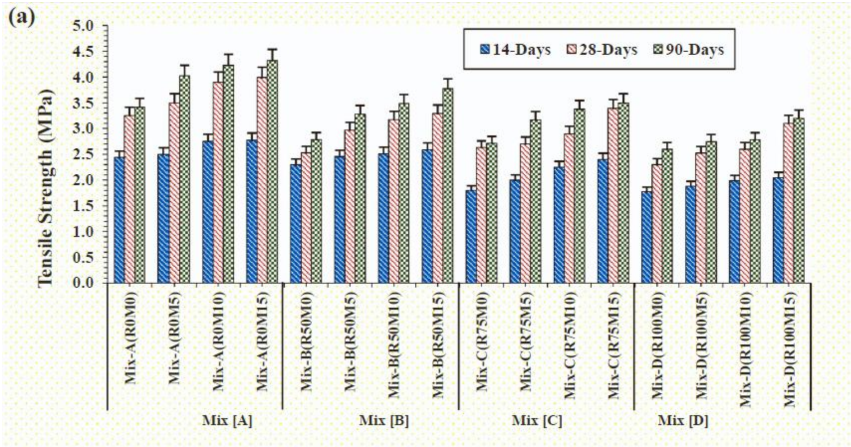


Figure 2.11: The Tensile Strength of Concrete with different Portion of Metakaolin used (Elhadi et al., 2023).

Moreover, metakaolin also reduced the water absorption in the concrete mixture. The measurement of water absorption is also a crucial parameter in structural concrete as it is directly correlated with the compressive strength of the concrete. Figure 2.12 shows that the inverse relationship between water absorption and compressive strength. The water absorption has decreased with higher compressive strength and durability. In another words, the higher percentage of metakaolin that has partially replaced the cement has resulted to a reduction of water absorption of concrete. It is because the pozzolanic reaction introduced by metakaolin was promoted the formation of insoluble hydration products within the concrete, it has effectively blocked the concrete pores and reduced the water absorption eventually. Additionally, the fine particle size of metakaolin contribute to a reduction in pore size, further improving the water resistance of concrete (Elhadi et al., 2023). Furthermore, the slump test that was conducted by Elhadi illustrated the decreases of slump values with the increases of metakaolin. This is due to the large surface area of the metakaolin exposed to surrounding allowed it to absorb water to occur the pozzolanic reaction.

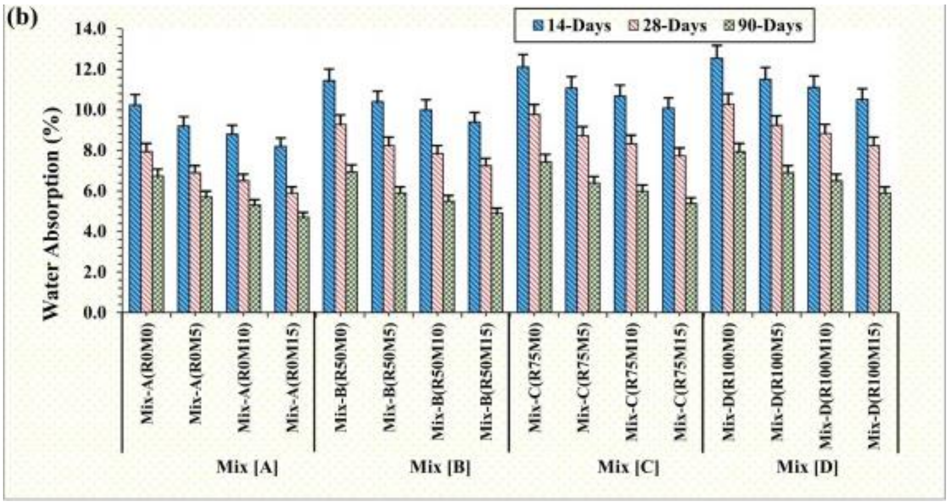


Figure 2.12: The Percentage of Water Absorption of Concrete with different Portion of Metakaolin used (Elhadi et al., 2023).

In summary, there is several advantages of the application of metakaolin in partially replacement of cement as shown in Figure 2.13. In term of environmental protection, usage of metakaolin able to reduce demand of the cement production and thereby reducing the CO_2 emissions. Furthermore, metakaolin also promoted the high-strength concrete, thermal resistant, corrosion resistant of concrete in order to protect the reinforcement bar inside the concrete and increase the durability.

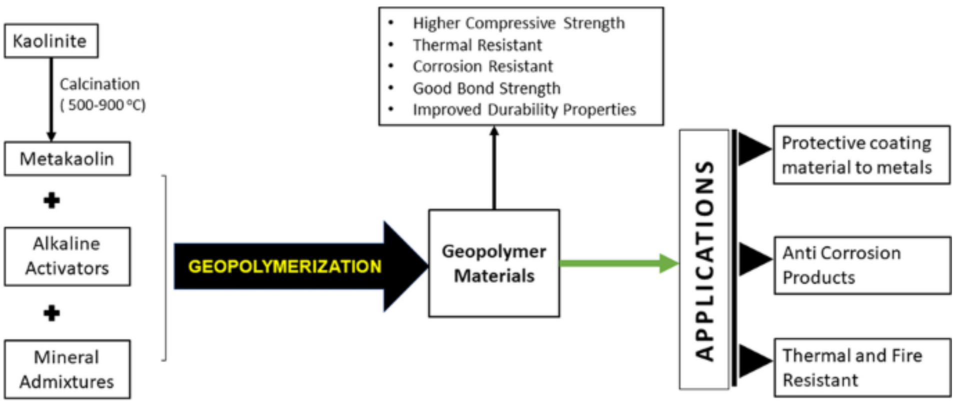


Figure 2.13: The Application of Metakaolin (Jindal et al., 2022).

2.5 Ordinary Portland Cement (OPC)

In the concrete mix design, OPC is an essential raw material or cementitious materials that is used to produce the conventional concrete or used as control concrete for concrete mix design experiment. There are various types of OPC are commercialized in the market, where they are mainly categorized into 5 classes in accordance with ASTM C150 (2007). They are Type I, Type II, Type III, Type IV a Type V. Where the Type I OPC is commonly used in general construction with the high C_3S content that can promote early strength. The Table 2.4 shows the chemical composition of the Type I Cement and contained highest percentage of CaO among the other compound.

Table 2.4: Chemical Composition of ASTM Class Type I Cement (Xie et al., 2019).

Compound	**Weight (%)**
CaO	63.82
SiO_2	20.09
Al_2O_3	3.87
SO_3	3.50
Fe_2O_3	1.69
MgO	2.22
Bogue Compositions	
C_3S	68.7
C_2S	5.80
C_3A	7.40
C_4AF	5.10

2.6 Aggregates

Based on the standard of ASTM C33/C33M (2018), aggregates are categorized into two main types, fine and coarse aggregates. Fine aggregates that can be found in market are natural sand, artificial sand or a combination of these materials. However, only the natural sand is used as fine aggregates for this study as shown in Figure 2.14. The standards stated that fine aggregates should be capable to pass through a 4.75 mm sieve and retained on a 0.0075 mm sieve. The application of fine aggregate is to fill up the gaps and void between cement particles and aggregate with larger size. On the other hand, the coarse aggregates are the particles that is unable to pass through or retained on a 4.75 mm sieve. Generally, coarse aggregates, such as limestones or gravels as shown in Figure 2.15. They have the average size of 40 mm are utilized for normal strength concrete which is between 20 to 40 MPa. While for the aggregates with the size of 19 mm can be used for high-strength concrete. In the concrete mixture process, the aggregates used up a significant portion with approximately 60 to 70 % of the overall concrete mixture content. Thus, a good quality aggregates is required to withstand compressive and tensile loads, promote toughness and free to impurities as possible.

Figure 2.14: Natural Sands as Fine Aggregate.

Figure 2.15: Limestone Gravels as Coarse Aggregates.

2.7 Microstructural Analysis

Microstructural analysis is conducted through the method of Scanning Electron Microscopy (SEM) analysis and Energy Dispersive X-ray (EDX) analysis to examine the microstructure of concrete in order to provide better insights into the performance of the concrete materials and deeper understanding of concrete properties and behaviour in accordance with the ASTM C 1723 (2016).

The research conducted by Abu-Saleem et al. (2021), the SEM tests were carried out to visualize on the interface transition zone between the recycled plastic aggregates, which are PET and HDPE plastic aggregate and the cementitious paste as shown in Figure 2.16 a) PET aggregates and b) HDPE aggregates. A gap of interface transition zone is observed due to the weak bonding between the plastic aggregates and cementitious waste as the plastic aggregates had a smooth surface texture.

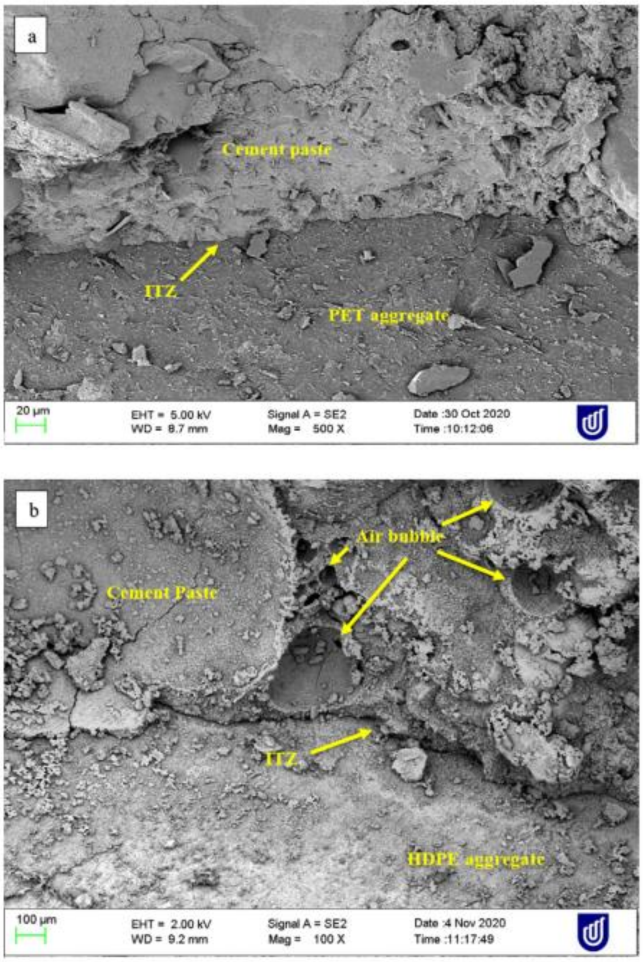


Figure 2.16: SEM Images of Concrete Mixture Containing Recycle Plastic Aggregates (Abu-Saleem et al., 2021).

On the other hand, according to the research conducted by Gopalakrishna and Pasla, (2023), SEM analysis was conducted on fly ash, metakaolin and GGBS using a Zeiss Merlin compact field emission SEM. However, in this chapter only show the part with metakaolin. Figure 2.17 demonstrates that the metakaolin particles have an angular and platy shape with an uneven surface. It is because the incineration process during the manufacturing of metakaolin. Additionally, the researcher has also performed the Energy Dispersive X-ray Spectroscopy (EDX/EDS) analysis for metakaolin to study the elements contained in metakaolin with weight percentages. Figure 2.17 illustrates the EDS analysis of weight percentage of metakaolin with different elements, which they are carbon (C), oxygen (O), Aluminium (Al) and Silicon (Si). Table 2.5 shows the detailed value of weight percentage of the metakaolin with elements. Neglect the oxygen and carbon, the metakaolin consisted of high weight percentage of Al and Si with the value of 11.98 and 12.22 respectively.

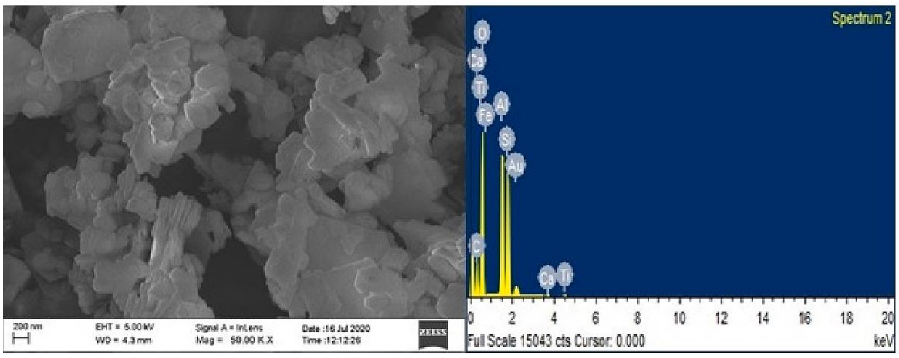


Figure 2.17: SEM and EDX Analysis of Metakaolin (Gopalakrishna and Pasla, 2023).

Table 2.5: EDX Analysis of Weight Percentage of Metakaolin with different Element (Gopalakrishna and Pasla, 2023).

Materials	Carbon	Oxygen	Aluminium	Silicon
Metakaolin	54.52	19.54	11.98	12.22

From the experiment conducted by (Li et al., 2022),the microstructure of the early stage of concrete mixture with various portions of metakaolin added is examined by using the method of SEM. In the Figure 2.18 a) shows occurring of some cement hydration products and obvious pores and cracks in the concrete mixture without metakaolin, indicated that the microstructure of the cement paste is loose, which can be adversely affected the concrete performance. However, with the increased proportions of metakaolin into concrete mixture from 5 % to 20 %, the occurrence of the pores and cracks were becoming lesser as the metakaolin was filled up the pores and promoted a denser and higher compressive strength of concrete.

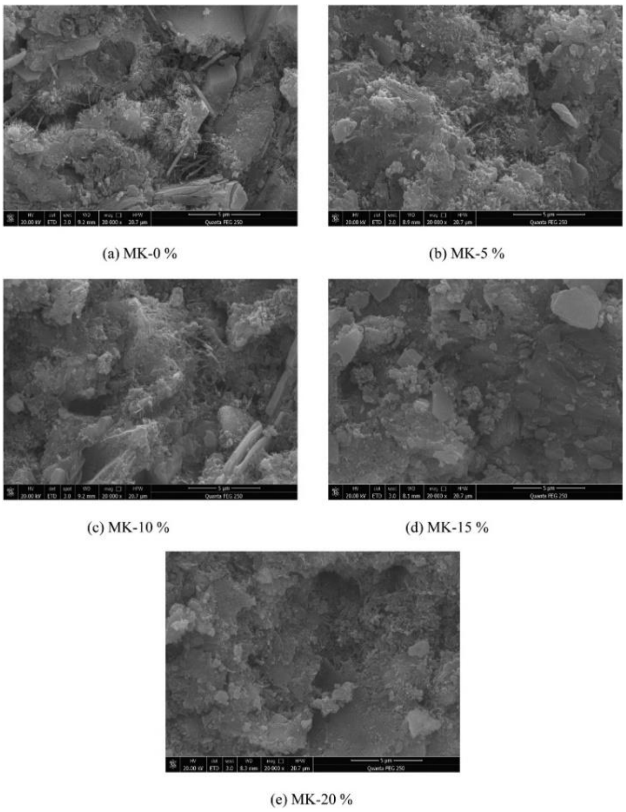


Figure 2.18: The Morphologies of Cement Paste with Different Metakaolin's Proportion (Li et al., 2022).

Lastly, X-Ray Diffraction (XRD) analysis is applied into the research to analyze the structure of the materials at the atomic and molecular levels. It functions by shining X-rays onto a specimen and measuring how they are scattered or diffracted by the atoms in the specimen, namely intensity against the degree of scanning with the minimum degree of 5 to maximum degree of 85. According to Dai et al. (2022), the study showed that in metakaolin, kaolinite that contained inside the metakaolin is a main mineral component and its characteristic peak 2θ were 12.3 %, 19.8 %, 24.9 % and 45.4 %, in addition of quartz, calcite, calcite and other minerals as shown in Figure 2.19.

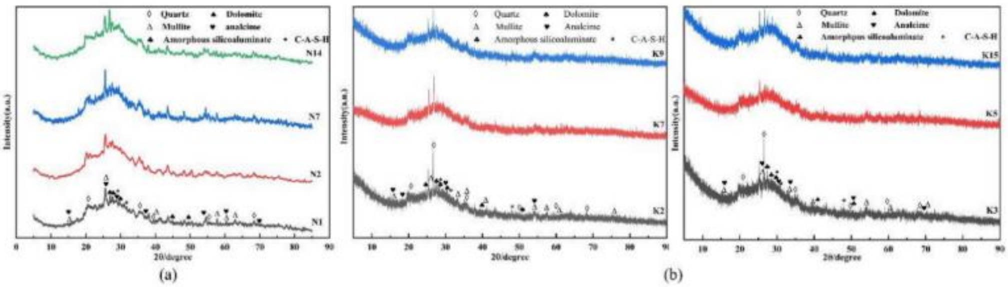


Figure 2.19: XRD Patterns of Geopolymers at 28 days: a) Na-Based Geopolymers and b) K-Based Geopolymers Dai et al., (2022).

2.8 Summary

In term of environmental protection, the recycled PET plastic waste is selected to be the sustainable materials that are used to replace the natural coarse aggregates in concrete mixture, such as limestone, in order to reduce the plastic waste pollution and improve the waste management system as well as the ease of obtained materials. The PET coarse aggregates added in concrete mixture has rather increase or decrease the workability in fresh stage but does not enhance the compressive strength of the concrete. Besides, of metakaolin is added in the concrete mixture to provide the opportunity to replace OPC to achieve the goals of CO_2 emissions reduction and high-strength concrete. To better understand the characteristics of the sustainable concrete mix, the microstructure of these raw materials is required to analyse by using the SEM method to obvious the composition of the components and the EDX method to analysis the element contained in these raw materials. Furthermore, XRD analysis is also use in this study to identify the chemical compound the concrete mix. The review on the articles is only either study the plastic waste substitution or metakaolin replacing the ordinary cement or no further microstructural analysis carry out. Therefore, in this study, two sustainable materials namely PET and metakaolin was used in the concrete mix design and study the fresh and hardened concrete properties as well as the microstructural analysis with application of SEM-EDX and XRD analysis.

CHAPTER 3

METHODOLOGY AND WORK PLAN

3.1 Introduction

This chapter presented the methodology of this experiment conducted. It described the complete process of producing a design concrete specimen, from preparation of raw materials to mixing procedures. The fresh and hardened properties tests of normal weight concrete specimens of 2400 kg/m^3 were carried out according to standards of BS EN and ASTM to ensure the consistency and precision of the data throughout the study.

3.2 Overall Flow Chart of the Experiment Works

Figure 3.1 shows the overall flow chart of the experimental works. Started with the section of introduction, literature review, material preparation and concrete casting, results and discussion and lastly conclusion and recommendations. In the section of material preparation and concrete casting, a trial mix was carried out for design concrete mixes with varying water to cement ratio to achieve a desired compressive strength of 25 MPa. Once an optimal design concrete mix was selected, actual mix was conducted and the fresh and hardened properties tests such as slump test, density determination, compressive and splitting tensile strength were conducted to determine the optimal concrete mix design with the integration of PET flakes and MK. Eventually, microstructural analyses namely SEM-EDX and XRD analysis were performed to study the chemical composition of the design concrete.

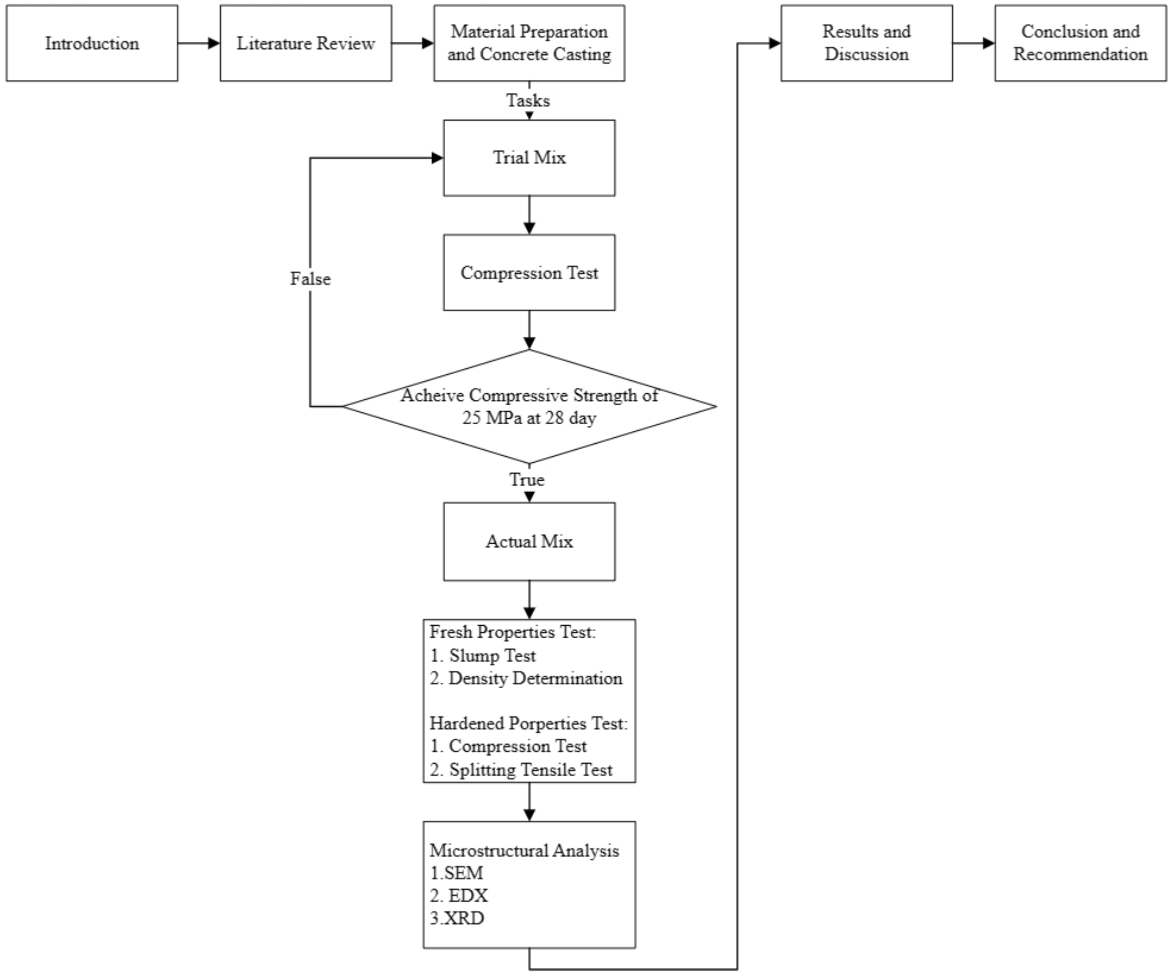


Figure 3.1: Flow Chart of Overall Work and Lab Experiment Procedure Detail.

3.3 Raw Materials

This section presented the raw materials used in the study, which is included Ordinary Portland Cement (OPC), coarse aggregates (CA) and fine aggregates, PET flakes, water, and Metakaolin (MK).

3.3.1 Ordinary Portland Cement (OPC)

The general cement that commonly used is the Type I Ordinary Portland Cement (OPC). Figure 3.2 shows the Orang Kuat, CEM I cement was manufactured by YTL cement, was used in this study. This branded cement has certified to MS EN 197-1:2014 with the grade of 52.5 kN. The OPC was sieved through a 600 μm sieve in accordance with ASTM 150 in order to sieve out the coagulated OPC. The chemical and physical properties of OPC was shown in Table 3.1. The extra OPC was kept in the airtight container after the concrete mixing to prevent the air exposure of OPC.

Figure 3.2: YTL "Orang Kuat" OPC.

Table 3.1: Chemical and Physical Composition of YTL "Orang Kuat" OPC.

Test		**Units**	**Specification MS EN 197-1: 2014 CEM I 42.5N**	**Test Results**
			Chemical Composition	
Insoluble Residue		%	≤ 5.0	0.4
Loss On Ignition (LOI)		%	≤ 5.0	3.2
Sulphate Content (SO_2)		%	≤ 3.5	2.7
Chloride (CI8*)		%	≤ 0.10	0.02
			Physical Properties	
Setting Time (Initial)		mins	≥ 60	130
Soundness		mm	≤ 10	1.2
Compressive Strength				
(Mortar Prism)	**: 2 days**	MPa	≥ 10	29.7
(1 : 3 : 0.5)	**: 28 days**	MPa	≥ 42.5 ; ≤ 62.5	48.9

3.3.2 Fine, Coarse Aggregates and PET Flakes

Firstly, the natural sands were used as fine aggregates in concrete mixture as shown in Figure 3.3. According to the ASTM C136, fine aggregates were passed through 4.75mm (No.4) and retained on a 150 µm (No.100) of the sieve level. Next, the limestone gravels are used as coarse aggregates as shown in Figure 3.4. The coarse aggregates were sieved through 20 mm, 10 mm and 4.75 mm. The particles that have passed through 4.75 mm sieve will not be used in accordance with ASTM 136, where it mentioned the coarse aggregates must be greater than 5 mm size. The sands and coarse aggregates were dried in oven for a day before the concrete casting. Both fine and coarse aggregates were later kept in clean and airtight container. PET flakes within the size range of 5 mm to 15 mm were used for partially replacement of coarse aggregates by percentages shown in Figure 3.5.

Figure 3.3: Natural Sands as Fine Aggregate.

Figure 3.4: Limestone Gravels as Coarse Aggregates.

Figure 3.5: PET Flakes.

3.3.3 Water

Tap water was used in this study to mix the concrete in accordance with ASTM C1602. Water must be clean and without any sediment or chemicals that used in concrete mix in order to perform the hydration process, resulting to the generation of calcium silicate hydrate (CSH) gel.

3.3.4 Metakaolin

Based on the Figure 3.6 shows the metakaolin was used in the concrete mixture to partially replace the OPC for specific purpose, such as improving the concrete properties of durability and strength. According to ASTM C618, metakaolin was classified as class N pozzolan specification. The metakaolin was carefully stored and the kept in the airtight container.

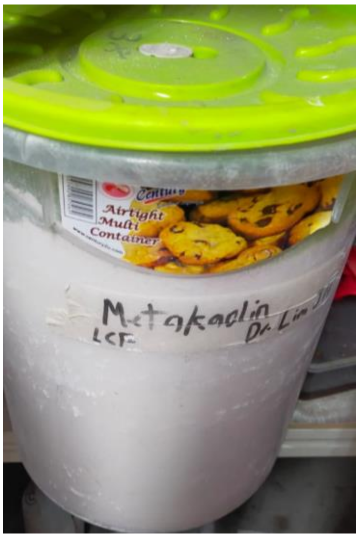


Figure 3.6: Metakaolin kept in Container.

3.4 Concrete Mould

Two types of the moulds were utilized to prepare the concrete specimens for different strength tests. Which they are cubic mould for compression test, cylinder mould for splitting test. According to the ASTM C31/C31M-19 and BS EN 12390-1, all the moulds' dimensions are standardized for concrete mix. Figure 3.7 shows the Cubic Mould and the Figure 3.8 shows Cylindrical Mould. Table 3.2 presents the moulds' dimensional for cubic and cylindrical specimens. Before concrete moulding, the internal surfaces of the moulds were required to clean and applied a layer of oil to ease the concrete demoulding.

Figure 3.7: Cubic Mould.

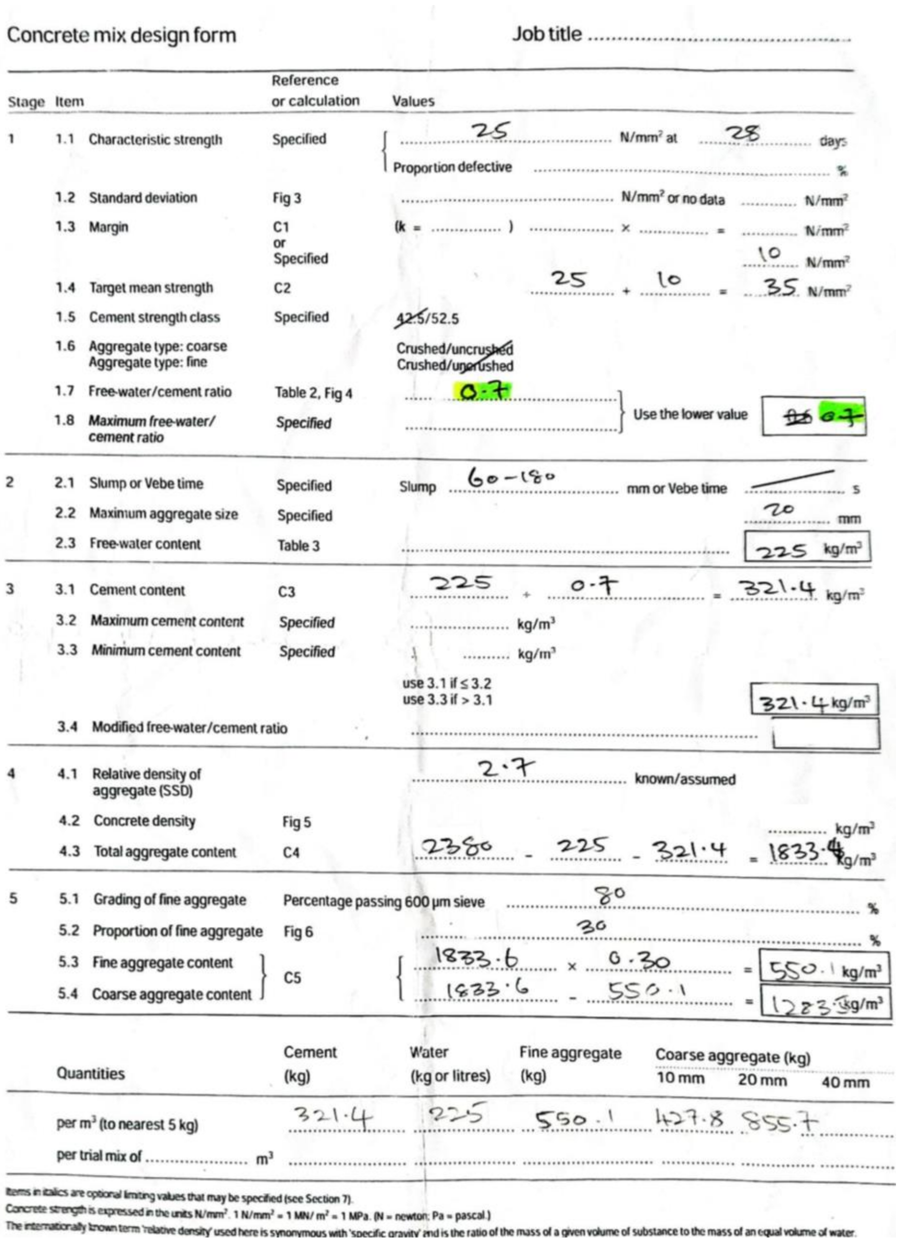
Figure 3.8: Cylindrical Mould.

Table 3.2: Summary of Mould Used.

Mould	Dimension (mm)	Strength Test
Cubic	$150 \times 150 \times 150$	Compressive Strength Test
Cylindrical	100×200	Splitting Tensile Strength

3.5 Trial Mix (Phase 1)

The trial mix is an important step to proceed before casting for an actual mix specimen in this study. A control mix concrete (CTR) and design concrete with 5 % PET replacement of coarse aggregates (PET-5) were prepared. A trial mix of CTR and PET-5 with only cubic specimen were required for preforming the compressive strength test for 7 days and 28 days It is because the concrete must have at least compressive strength of 25 MPa at 28 days to proceed to actual mixes. The trial mix was carried out with various water to cement ratio of CTR and PET-5 from 0.6 to 0.7 with an interval of 0.05. After results of the compressive strength were recorded at 7 days and 28 days, an optimal water to cement ratio was selected for actual concrete mix design. Figure 3.9 shows the concrete mix design with water / cement ratio of 0.7. The slump value was set to be 60 to 180 mm as per standard. Eventually, the quantities for cement, water, fine aggregates, 10 mm and 20 mm of coarse aggregates were 3214 kg / m^3, 225 kg / m^3, 550.1 kg / m^3, 427.8 kg / m^3 and 855.7 kg / m^3 respectively.



Concrete mix design form　　　Job title ....

Stage	Item	Reference or calculation	Values
1	1.1 Characteristic strength	Specified	25 N/mm² at 28 days; Proportion defective ..... %
	1.2 Standard deviation	Fig 3	..... N/mm² or no data ..... N/mm²
	1.3 Margin	C1 or Specified	(k = ...) ..... × ..... = ... N/mm²; 10 N/mm²
	1.4 Target mean strength	C2	25 + 10 = 35 N/mm²
	1.5 Cement strength class	Specified	~~42.5~~/52.5
	1.6 Aggregate type: coarse Aggregate type: fine		Crushed/~~uncrushed~~ Crushed/~~uncrushed~~
	1.7 Free-water/cement ratio	Table 2, Fig 4	0.7
	1.8 *Maximum free-water/cement ratio*	*Specified*	..... Use the lower value ~~0.6~~ 0.7
2	2.1 Slump or Vebe time	Specified	Slump 60–180 mm or Vebe time — s
	2.2 Maximum aggregate size	Specified	20 mm
	2.3 Free-water content	Table 3	225 kg/m³
3	3.1 Cement content	C3	225 ÷ 0.7 = 321.4 kg/m³
	3.2 *Maximum cement content*	*Specified*	..... kg/m³
	3.3 *Minimum cement content*	*Specified*	..... kg/m³; use 3.1 if ≤ 3.2; use 3.3 if > 3.1; 321.4 kg/m³
	3.4 Modified free-water/cement ratio		.....
4	4.1 Relative density of aggregate (SSD)		2.7 known/assumed
	4.2 Concrete density	Fig 5	..... kg/m³
	4.3 Total aggregate content	C4	2380 − 225 − 321.4 = 1833.6 kg/m³
5	5.1 Grading of fine aggregate	Percentage passing 600 µm sieve	80 %
	5.2 Proportion of fine aggregate	Fig 6	30 %
	5.3 Fine aggregate content	C5	1833.6 × 0.30 = 550.1 kg/m³
	5.4 Coarse aggregate content	C5	1833.6 − 550.1 = 1283.5 kg/m³

Quantities	Cement (kg)	Water (kg or litres)	Fine aggregate (kg)	Coarse aggregate (kg) 10 mm	20 mm	40 mm
per m³ (to nearest 5 kg)	321.4	225	550.1	427.8	855.7	
per trial mix of ..... m³						

Items in italics are optional limiting values that may be specified (see Section 7).
Concrete strength is expressed in the units N/mm². 1 N/mm² = 1 MN/m² = 1 MPa. (N = newton; Pa = pascal.)
The internationally known term 'relative density' used here is synonymous with 'specific gravity' and is the ratio of the mass of a given volume of substance to the mass of an equal volume of water.

Figure 3.9: Concrete Mix Design with W/C of 0.7

3.6 Sieve Analysis (ASTM C136/C136M-19)

In this study, sieve analysis was performed for fine and coarse aggregates and PET flakes as outlined in ASTM C136/C136M standard. Thus, 500 g of fine aggregates, 1000g coarse aggregates and PET flakes were prepared. It is a method to determine the particle size distribution of fine and coarse aggregates. This is important in ensuring that aggregates meet the specified grading as per required for various construction purposes. The process involved the aggregates that passing through a series of sieves of smaller mesh sizes and the amount of retained aggregates on each sieve was weighed and recorded. Figure 3.10 shows the machine for fine aggregates sieve analysis and Figure 3.11 shows the machine for coarse aggregates sieve analysis.

Figure 3.10: Machine for Fine Aggregates Sieve Analysis.

Figure 3.11: Machine for Coarse Aggregates Sieve Analysis.

3.7 Actual Mix

In this study, there are 2 Phases needed to be performed for actual mix and several tests were conducted such as slump test for workability, density determination, compressive strength test and splitting tensile strength test in order to investigate the effect and correlation of PET and metakaolin substitution in normal concrete. The content of fine aggregates and water were set to be constant. By obtaining the of optimal design concrete mix proportions from trial mix, a concrete mix proportion for PET flakes replacement of coarse aggregates with varying percentages from 5 % to 15 % with an interval of 5 % was prepared in Phase 2 with the cubic and cylindrical specimens. They are PET-5, PET-10 and PET-15 respectively. Afterward, an optimal design concrete mix in Phase 2 was selected in term of compressive strength recorded and was applied in Phase 3. In phase 3, optimal design concrete mix was used with different percentage of metakaolin substitution of OPC from 5 % to 10 % with an interval of 2.5 %.

3.8 Concrete Mix Procedure and Curing

To produce a normal concrete, the preparation and weighing of the raw materials was performed namely OPC, water, sands, limestone gravels with weighing machine as shown in Figure 3.12. Next, dry mix of the raw materials evenly is carried out by using the mixing machine as shown in Figure 3.13. The raw materials that were included are OPC, sands and limestones. The water was then slowly added into the dry mix allowed the even concrete mix and continue operating machine for 10 to 15 minutes. Before pouring the fresh stages of the concrete into the mould, the fresh properties test was carried out to obtain the slump test and fresh density. Afterward, fresh concrete was poured into six 150 x 150 x 150 mm dimension of cubic moulds and six 100 x 200 mm dimension of cylindrical moulds in 3 layers, each layer consisted of one third of the cubic or cylindrical moulds’ volume. A long steel tamping rod was used to tap the fresh concrete for 25 times in order to release the air voids and prevent segregation. The process of concrete hardening and settling required at least a day to complete as shown in Figure 3.14. After demoulding of the specimens by using the air blower for cubic mould and unscrewed for cylindrical mould, the curing of the specimens is carried out by submerging the specimens into the water for 7 days and 28 days as shown in Figure 3.15.

Figure 3.12: Raw Materials Preparation after Weighing Machine.

Figure 3.13: Dry Mix in Mixing Machine.

Figure 3.14: Hardening and Settling of Concrete Mix.

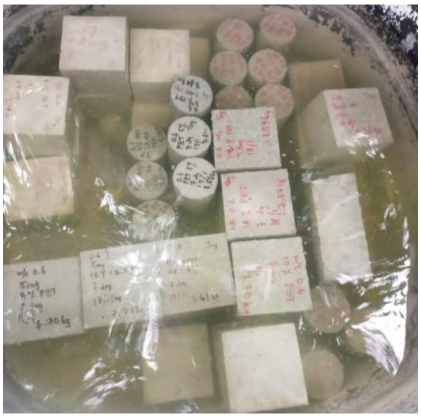


Figure 3.15: Water Curing.

3.9 Fresh Concrete Properties Test

This test was conducted to study the workability of the concrete. A slump test was conducted in this study.

3.9.1 Slump Test (ASTM C143/C143M)

The slump test was conducted to determine the workability of the fresh concrete in accordance with ASTM C143/C143M. Figure 3.16 shows the slump cone was prepared for slump test. After the fresh concrete is produced, a slump cone was placed on flat surface and fresh concrete was filled into cone in three layers, each layer consisted of one third of the slump cone's volume. The long steel tamping rod was used to tap the fresh concrete for 25 times to release the air bubbles and evenly distribute the fresh concrete. Once the fresh concrete has reached to the top of the slump cone, the excessive fresh concrete was removed and the cone was lifted up vertically and slowly. The height of the slumped fresh concrete has recorded by using the meter ruler for the study of workability as shown in Figure 3.17.

Figure 3.16: Slump Cone for Slump Test.

Figure 3.17: Slump Values of Fresh Concrete.

3.10 Hardened Concrete Properties Test

There are two methods of hardened concrete test, which are the destructive and non-destructive methods. In this study, only the destructive method is implemented. Typically, compression test, splitting tensile test and flexural test are the destructive methods that were utilized in this study.

3.10.1 Compressive Strength Test (BS EN 12390-3)

The compressive strength test was carried out to obtain the compressive strength of cubic specimens at both 7 days and 28 days. A universal compression machine was used to run the test. The cubic specimens were oven-dried after taken from water curing to record the saturated concrete weight. Once the machine was setup completely, cubic specimen with the smooth surface was subjected to place to the surface of compression and adjusted to the centre of the machine's base plate as shown in Figure 3.18. Eventually, the compressive strength was calculated based on the BS EN 12390-3. Expressed as:

$$f_c = \frac{F}{Ac} \tag{3.1}$$

Where,

fc = Compressive Strength, *MPa*

F = *Peak Load, N*

Ac = *Cross-sectional area of cubic specimen,* m^2

Figure 3.18: Compression Test of Cubic Specimen.

3.10.2 Splitting Tensile Strength Test (ASTM C496)

The splitting tensile strength test was conducted to obtain the splitting tensile strength of the cylindrical specimens for both 7 days and 28 days. A universal compression machine is utilized to carry out the test as shown in Figure 3.19. The cylindrical specimens were oven-dried after air blowing on the surface of the specimens to record the saturated concrete weight. A packing strips were placed at the top and the bottom of the specimen that was subjected to place to the surface of compression and adjusted to the centre of the machine's base plate. Finally, the splitting tensile strength was calculated based on ASTM C496. Expressed as:

$$T = \frac{2P}{\pi LD} \tag{3.2}$$

Where,

T = Splitting tensile strength, *MPa*

P = *Peak Load*, *N*

L = *Cylindrical specimen's length*, *mm*

D = *Cylindrical diameter*, *mm*

Figure 3.19: Splitting Tensile Test of Cylindrical Specimen.

3.11 Microstructural Analysis

The microstructural analysis performed by using the method of scanning electron microscope (SEM) to obvious the characteristic of the concrete's surface and Energy Dispersive X-ray (EDX) to analyse the elements in the designed concrete. Before conducting the SEM-EDX analyses, the specimens were required to place into the Emitech SC7620 Coater with Vacuum Pump in order to apply a thin coating layer of gold or palladium onto the specimens as shown in Figure 3.20.

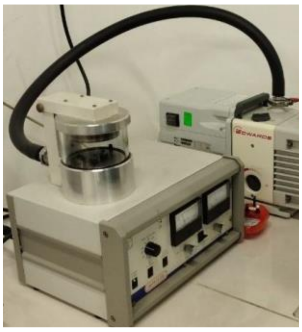


Figure 3.20: Emitech SC7620 Coater Machine.

3.11.1 Scanning Electron Microscope (SEM) (ASTM C1723)

Figure 3.21 shows the machine that was operated to carried out the microstructure analysis through both methods of SEM and EDX. According to ASTM C1723, the SEM method is a powerful imaging tool that was used in this study to analyse the microstructure of the destructed specimen more detail and visualize by the emission of electrons and photons onto the surface of the specimen. In that case, the SEM method was used to observe and analyse the interaction among the elements of the specimen.

Figure 3.21: Hitachi S-3400N SEM Machine.

3.11.2 Energy Dispersive X-ray (EDX) (ASTM C1723)

While the EDX was used in this study to qualitatively or quantitatively determine the elemental composition of very small in volume intersection the surface of the observed destructed specimen and those measured compositional determinations can be correlated with specific features that was observed in the SEM image.

3.11.3 X-Ray Diffraction (XRD) (ASTM E3294-22)

Figure 3.22 shows the X-ray diffraction (XRD) that was used to identifying the crystalline components within a sample and carried out quantitative and qualitative phase analyses. The scanning range for this study was from minimum 5º to 85ºmaximum, where the scanning speed was 2º/min. Thus, the duration to conduct the XRD was 40 minutes for a sample.

Figure 3.22: X-ray Diffraction Machine.

3.12 Summary

In conclusion, this chapter introduced the raw materials that were used in concrete mixture. They are OPC, natural sands and coarse aggregates, PET flakes, water and metakaolin. In the design concrete mix proportion of normal weight concrete, the sands and water content were fixed, while PET and metakaolin content were varying with 5%, 10% and 15% for PET substitution and 5 %, 7.5 % and 10 % for metakaolin. The aim of this study was to achieve the concrete's compressive strength of 25 at 28 days with varying proportions of PET and metakaolin. The mixing procedure and curing were descripted in detail. Fresh properties test and hardened properties such as slump test, density determination, compressive and splitting tensile strength test were carried out to study the effect of PET and metakaolin inside the concrete mixes on workability and hardened properties of the concrete. Lastly, the microstructure analysis is performed with the SEM, EDX and XRD method to visualize the characteristics of concrete surface and elemental composition of concrete.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter discussed on the mix proportions and compressive strength of two trial concrete mixes (Phase 1). They are trial control concrete mix (CTR) and trial concrete mix with 5 % PET replacement of coarse aggregates (PET-5). Following the determination of the optimal concrete mixture of two trial concrete mixes based on the compressive strength results obtained, subsequent experimental tests and analyses will be carried out in real mix phase. This phase involved replacement of coarse aggregates (CA) with varying percentages of PET (Phase 2) and an optimal design concrete mix with PET flakes were selected for further experimental works of varying amount of metakaolin (MK) replacing OPC (Phase 3). The effects of PET and MK replacements will be thoroughly examined, leading to the eventual selection of the optimal design concrete mix. Moreover, the microstructural analysis was performed to further understand the chemical composition of the optimal designed concrete.

4.2 Sieve Analysis

According to ASTM C136/C136M-19 standard, a grading process namely sieve analysis was performed to determine the particle size distribution of coarse and fine aggregates. 1000 grams of limestone, 1000 g of PET flakes and 500 grams of sand are required to perform the test. There are 5 levels of sieves opening was arranged in descending order from 20 mm to 4.75mm for coarse aggregates whereas 7 levels of sieve opening were arranged in descending order from 4.75 mm to 150 μm, ending with flat pan for fine aggregates. The cumulative percentage method is applied by measuring and recording the mass of coarse aggregates and sand particles retained on each sieve level and converted it to percentage. Therefore, the cumulative percentage retaining and passing of coarse and fine aggregates were computed in Table 4.1, Table 4.2 and Table 4.3 respectively.

Moreover, the fineness modulus (FM) of coarse and fine aggregates was determined to identify the average size of the particles in coarse and fine aggregate by an index number. Based on the ASTM C33 standard, the fineness modulus of coarse aggregate is 5.5 to 8.0 whereas fineness modulus of fine aggregate is between 2.3 and 3.1. Where the formula of FM is as shown below:

$$Fineness\ Modulus\ (FM) = \frac{N(100) - Sum\ Total\ of\ Passing\ (\%)}{100} \quad (4.1)$$

Where,

N = Number of sieves involved in the sum total of percent passing from largest sized noted to and including the No.100 (150µm)

Thus, the FM of modulus (FM) values of limestone, PET flakes and sand were 7.11, 6.99 and 2.38 respectively, which was falls within the required range. FM of 7.11 for coarse aggregates indicated that the average particle size is in between 7th and 8th sieves which is in the range from 10 mm to 20 mm. Whereas PET flakes have the FM values of 6.99 ≈ 7.0, thus the average particle size of PET flakes are around 10 mm. Other than that, FM of 2.38 for fine aggregates represented that the average particle size is in between 2nd and 3rd sieves which is in between 300µm and 600 µm. Figure 4.1 shows the distribution of fine, coarse aggregates and PET flakes with the percentage of finer aggregates passing against the sieve size (mm). Based on the result computed, the limestone, PET flakes and sand sample were suitable for concrete mix.

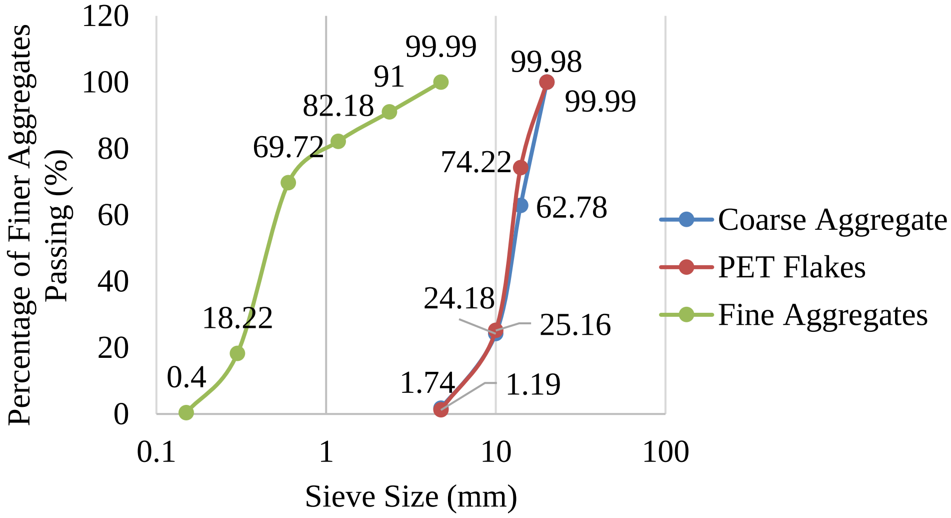


Figure 4.1: Distribution of Aggregates Particles Size.

Table 4.1: Sieve Analysis Results of Coarse Aggregates.

Sieve Sizes	Weight				Cumulative Percentage	
	Empty Sieve (g)	**Sieve + Aggregates Retained (g)**	**Aggregates Retained (g)**	**Aggregates Retained (%)**	**Coarser (%)**	**Finer (%)**
20 mm	391.4	391.6	0.2	0.02	0.02	99.98
14 mm	398.0	750.4	372.0	37.20	37.22	62.78
10 mm	451.4	800.9	386.0	38.60	75.82	24.18
4.75 mm	489.2	703.8	224.6	22.46	98.26	1.74
Pan	245.0	263.2	16.9	1.69	100	0.00
		Total	**999.7**	**100**		

Table 4.2: Sieve Analysis Results of PET Flakes.

Sieve Sizes	Weight				Cumulative Percentage	
	Empty Sieve (g)	Sieve + Aggregates Retained (g)	Aggregates Retained (g)	Aggregates Retained (%)	Coarser (%)	Finer (%)
20 mm	391.4	391.4	0.0	0.0	0.00	100
14 mm	398.0	705.8	257.8	25.78	25.78	74.22
10 mm	451.4	942.5	490.6	49.06	74.84	25.16
4.75 mm	489.2	728.9	239.7	23.97	98.81	1.19
Pan	245.0	256.9	11.9	1.19	100	0.00
		Total	**1000**	**100**		

Table 4.3: Sieve Analysis Results for Fine Aggregates.

Sieve Sizes U.S. Series	**Weight**				**Cumulative Percentage**		**ASTM C33-13 Limit For Fine Aggregates (%)**
	Empty Sieve (g)	**Sieve + Aggregates Retained (g)**	**Aggregates Retained (g)**	**Aggregates Retained (%)**	**Coarser (%)**	**Finer (%)**	
No.4 (4.75 mm)	490	490	0	0.00	0	100	95 to 100
No.8 (2.36 mm)	470	514.91	44.91	9.00	9.00	91.00	80 to 100
No.16 (1.18 mm)	370	414.01	44.01	8.82	17.82	82.18	50 o 85
No.30 (600 μm)	340	442.09	102.09	20.46	30.28	69.72	25 to 60
No.50 (300 μm)	370	587.07	217.07	43.50	81.78	18.22	5 to 30
No.100(150 μm)	370	458.92	88.92	17.82	99.60	0.40	0 to 10
Pan	250	252.00	2.00	0.40	100	0	0 to 3
		Total	**499**	**100**			

4.3 Trial Mix (Phase 1)

This chapter provides insights into the mix proportions and compressive strength of two trial concrete mixes. They are trial control concrete mix (CTR) and trial concrete mix with 5% Polyethylene Terephthalate (PET) replacing the coarse aggregates (PET-5) under varying water to cement ratio. Once the optimal mixture of two trial concrete mixes has been determined based on the compressive strength results obtained, subsequent experimental tests and analyses will be conducted in real mix phase.

4.3.1 Trial Control Concrete Mix

The trial mix of control concrete (CTR) is prepared with the basic materials namely Ordinary Portland Cement (OPC), find aggregates, coarse aggregates and water. The concrete mix is conducted under three different water to cement ratio. According to the standard ASTM C143/C143 M, the slump value is acceptable in between the range of 60 mm to 180 mm for normal concrete mix. Based on the Table 4.4 shows the design of mix proportion for the trial mix of CTR with water to cement ratio of 0.6, 0.65 and 0.70. The values of the mix proportions are based on 1 m^3 of concrete volume by using absolute method.

Table 4.4: Design Mix Proportions of Trial Mixes of CTR.

W/C Ratio	**Unit Weight (kg/m^3)**			
	OPC	**Fine Aggregates (<4.75 mm)**	**Coarse Aggregates**	
			10 mm Aggregates	**20 mm Aggregates**
0.60	375.0	534.0	415.3	830.7
0.65	346.2	542.7	422.1	844.2
0.70	321.4	550.1	427.8	855.7

Firstly, the fresh properties test of the trial control mix is carried out by implementing the slump test to determine the workability of the fresh concrete. Figure 4.2 shows the slump values of w/c ratios 0.6, 0.65 and 0.70 are 135 mm, 148mm, and 170 mm respectively. The results obtained indicates that workability of fresh concrete is gradually increased as the w/c ratio increased. It is because of the water serves as a lubricating agent among the cement particles, aggregate and other elements within the concrete blend, aiding in friction reduction and enhancing the flowability of fresh concrete, while at the same time contributing to its cohesion by binding the particles together.

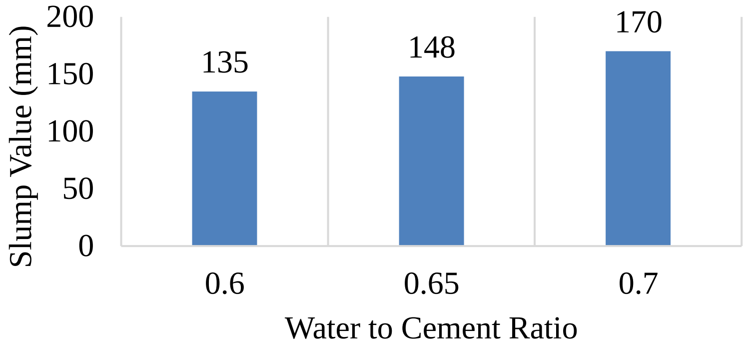


Figure 4.2: Slump Test of Trial Mix CTR with various W/C Ratio.

Furthermore, fresh and hardened density of trial CTR with different W/C ratio were recorded and demonstrated in Figure 4.3. In the figure showed that the lower the W/C ratio leaded to greater fresh and hardened density of the concrete as more water promotes porosity, shrinkage when hardening. The hardened concrete has decreased in density compared to fresh concrete as the small pores and voids are formed during the hydration process.

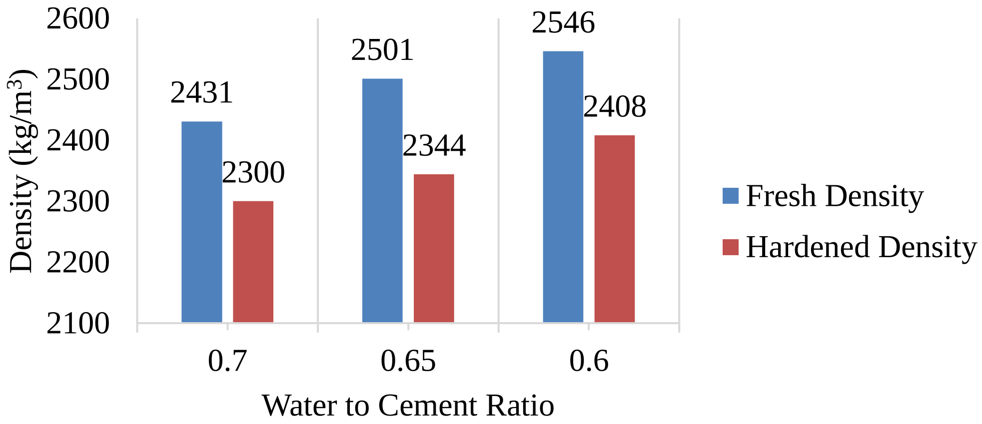


Figure 4.3: Fresh and Hardened Density of Trial Mixes of CTR.

Moreover, the compressive strength of trial mix of cubic specimens for CTR were obtained by conducting the compressive strength test of 7 days and

28 days curing of trial mixes of CTR. Figure 4.4 demonstrates that the Compressive Strength of the CTR is increased when the W/C ratio is decreased as more water leads to less effective binding od particles. All the CTR under 0.70, 0.65 and 0.60 are achieved the desired strength of 25 MPa minimum at 28 days. However, it is expected that the replacement of coarse aggregates with PET flake will be dramatically affected the compressive strength of the concrete negatively. Thus, the further trial mix was carried out with minimum 5 % PET replacement of coarse aggregates and will be discussed in 4.3.2 subtitle.

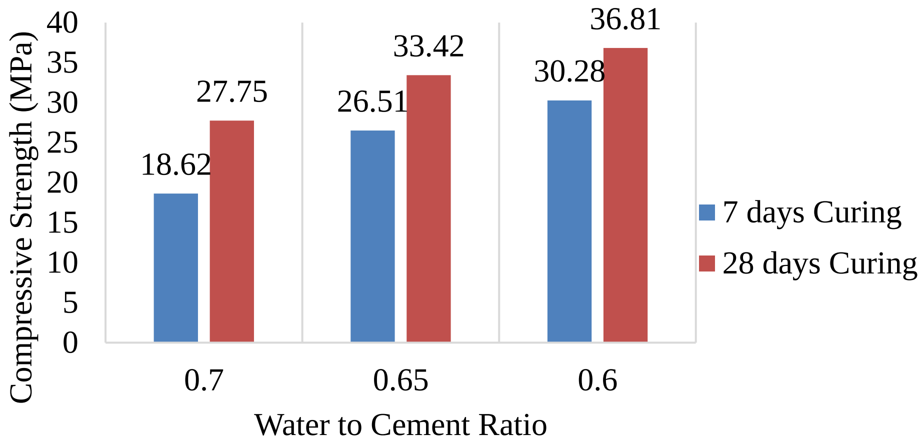


Figure 4.4: Compressive Strength (MPa) of Trial Mixes of CTR.

4.3.2 Trial Mix with 5% PET Replacement of Natural Coarse Aggregate

The trial concrete mixes with 5 % PET replacement of natural coarse aggregates (PET-5) were prepared. The volumetric method is applied to partially substitute the unit weight of natural coarse aggregates with PET flakes as the PET flakes are the lightweight coarse aggregates with the specify gravity of 1.35 while the standard specific gravity of limestone coarse aggregates is 2.65. The formula for the calculation of the trial mix with 5 % PET flakes is shown below. Thus, the mix proportions of trial mix of PET-5 under different water to cement ratio has prepared as shown in Table 4.5.

$$5\ \% \times \frac{Unit\ Weight\ of\ Natural\ Coarse\ Aggregates}{specify\ gravity\ of\ Natural\ Coarse\ Aggregates\ \times 1000} = \frac{Unit\ Weight\ of\ PET\ Flakes}{specify\ gravity\ of\ PET\ Flakes \times 1000} \tag{4.2}$$

Where,

specify gravity of natural coarse aggregate = 2.65

specify gravity of PET flakes = 1.35

Table 4.5: Design Mix Proportions of Trial Mixes of PET-5.

W/C Ratio	Unit Weight (kg/m^3)				
	OPC	Fine Aggregates (<4.75 mm)	Coarse Aggregates		
			10 mm Aggregates	20 mm Aggregates	5 % PET
0.60	375.0	534.0	394.44	789.44	30.16
0.65	346.2	542.7	401.00	802.00	32.25
0.70	321.4	550.1	406.41	812.92	32.69

First of all, the fresh properties test of the trial mixes of PET-5 namely slump test is conducted to identify the workability of the fresh concrete. Figure 4.5 shows the slump values of W/C ratios 0.6, 0.65 and 0.70 are 113 mm, 137 mm, and 160 mm respectively. Similarly, greater the W/C ratio leads to greater slump values. However, the slump value was decreased after partially replacement of coarse aggregate with PET as their porous nature allows them to absorb more water and reduced the amount of water available for hydration of cement paste, thus, the slump value is lower compared to the trial mix CTR.

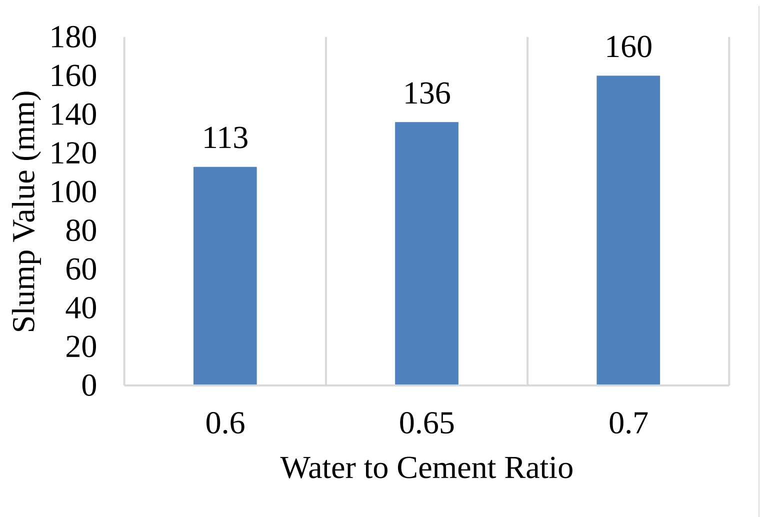


Figure 4.5: Slump Test of Trial Mix PET-5 with Various W/C Ratio.

Subsequently, fresh and hardened density of trial mixes of PET-5 were obtained and illustrated in Figure 4.6. The result showed that the fresh and hardened density of trial mixes of PET has decreased in overall density compared to the CTR as PET flakes are typically lighter than natural coarse aggregates. Moreover, PET flakes acted as waterproof materials that has trapped the air void formed in the process of hydration and also prevented hydration reaction between cement and water, which caused the concrete has dramatically drops in hardened density from fresh density compared to trial mixes CTR.

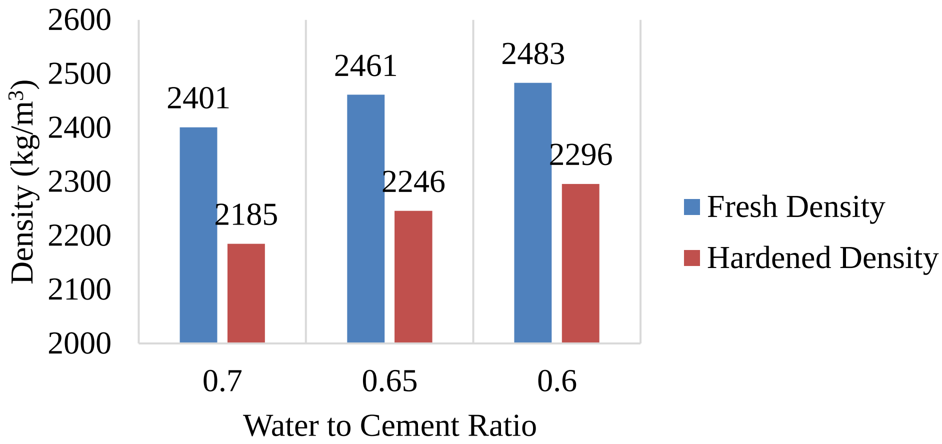


Figure 4.6: Fresh and Hardened Density of Trial Mixes of PET-5.

Based on the Figure 4.7 shows the compressive strength of 7 days and 28 days of trial mix with PET-5. The trial mix with PET-5 has increased in compressive strength when the W/C ratio is decreased. However, overall compressive strength of trial mixes of PET-5 has slightly lower compared to the compressive strength of trial mixes of CTR as the bonding between PET flakes and cement paste is weaker than the bonding between the natural coarse aggregates and cement during the hydration process. The results obtained showed that the trial mix with PET-5 under 0.6 W/C ratio with compressive strength of 32.82 MPa was achieved the desired compressive strength of 25 MPa on 28 days. Whereas the compressive strength of trial mix with PET-5 under 0.70 and 0.65 W/C ratio has 23.83 MPa and 24.91 MPa respectively, which does not achieve the requirement.

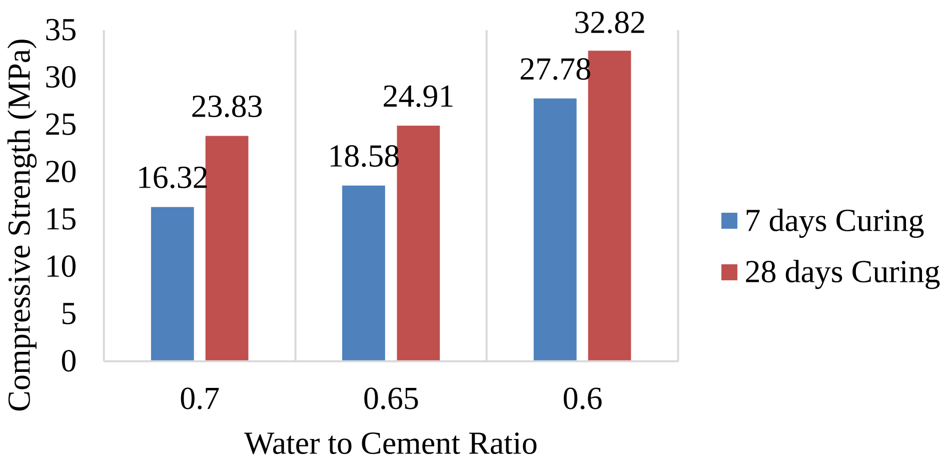


Figure 4.7: Compressive Strength (MPa) of Trial Mixes of PET-5.

4.3.3 Summary

In conclusion, the sieve analysis test has carried out to determine the particle sizes distribution. The average particle sizes of limestone, PET flakes and sand were 10 mm to 20 mm, 10 mm and 300 μm to 600 μm respectively. Among the trial mixes, CTR and PET-5 with the W/C ratio of 0.60 have satisfactory to the minimum 28 days compressive strength of 25 MPa. Thus, these mix proportions were selected as optimal mix proportions. The optimum design mix proportions were adopted for further experimental test namely compressive strength and splitting tensile strength.

4.4 Actual Mix with PET Flakes Replacement (Phase 2)

This chapter explored the effect of incorporating PET flakes of varying intensities to replace the coarse aggregates (CA) in normal concrete under W/C ratio of 0.6. The experimental works included fresh concrete test namely slump test and density determination, as well as hardened concrete test such as compressive strength and splitting tensile strength. A control normal concrete (CTR) and three concrete mixes with PET flakes replacing CA in 5 % intervals are prepared. The optimal mix proportion is selected for further adoption in Phase 3: Actual Mix with MK Powder Replacement of OPC. Table 4.6 shows the design mix proportions of CTR and PET with 5 %, 10 % and 15 % of PET flakes replacing the CA, there were PET-5, PET-10 and PET-15 respectively.

Table 4.6: Design Mix Proportions of CTR, PET-5, PET-10 and PET-15.

Design Mix	**W/C Ratio**	**Unit Weight (kg/m^3)**				
		OPC	**Fine Aggregates (<4.75 mm)**	**Coarse Aggregates**		
				10 mm Aggregates	**20 mm Aggregates**	**PET Flakes**
CTR	0.6	375.0	534.0	415.3	830.7	0.0
PET-5	0.6	375.0	534.0	394.4	789.4	30.2
PET-10	0.6	375.0	534.0	373.6	747.6	63.5
PET-15	0.6	375.0	534.0	352.9	706.0	95.2

4.4.1 Fresh Properties

The fresh properties included the slump test of CTR and replacement of CA with 5 %, 10 % and 15 % of PET flakes. Figure 4.8 shows the increased in PET flakes replacement gradually reduced the slump value of concrete mixes. The results obtained was similar to the experiment carried out by the (Saikia and De Brito, 2014), the experiment explained the decreased in slump value with the addition of PET coarse aggregates is due to their sharper edges compared to natural CA. PET coarse aggregates also differ from natural CA in their angular and non-uniform nature and the flaky composition.

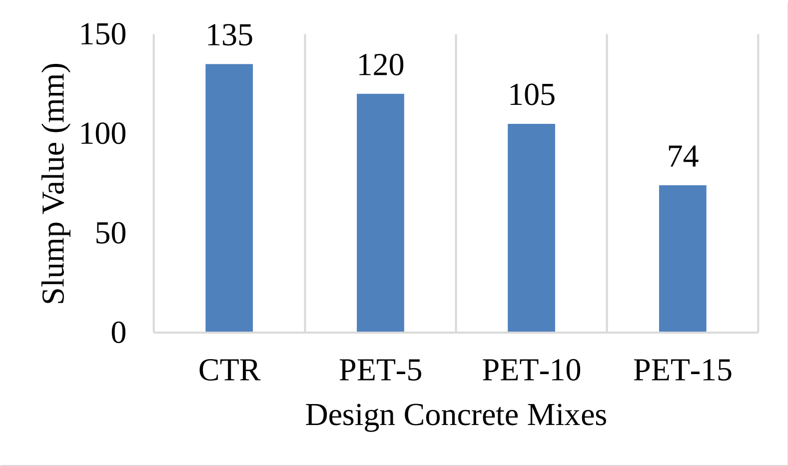


Figure 4.8: Slump Values of Various Concrete Mixes.

4.4.2 Density

Figure 4.9 illustrates the fresh and hardened density of various design concrete mixes. The overall hardened density of concrete mixes was lower than fresh density of concrete mixes. Overall density was gradually decreased by around 1 % with increased in PET flakes replacement compared to the CTR as the unit weight of PET flakes were lower than the CA that contributed to this phenomenon.

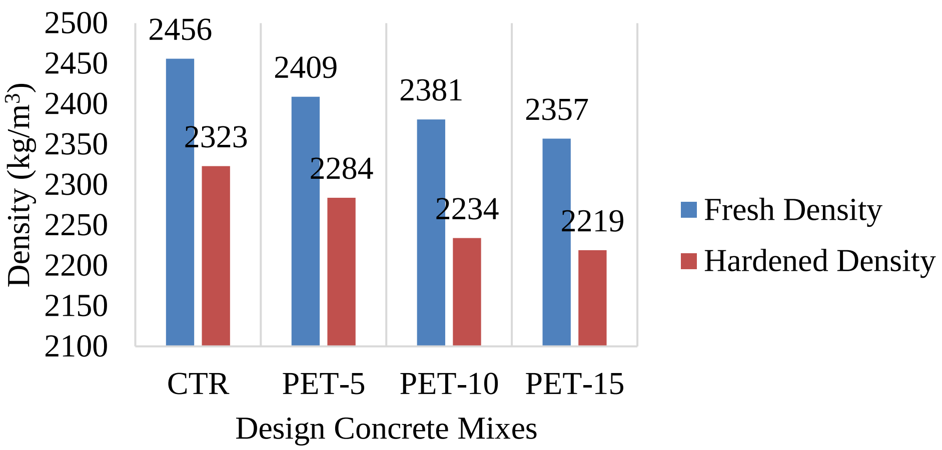


Figure 4.9: Fresh and Hardened Density of Various Concrete Mixes.

4.4.3 Compressive Strength

The compressive strength of CTR, PET-5, PET-10 and PET-15 for 7 days and 28 days were obtained and recorded in Figure 4.10. It was illustrated the compressive strength trend of concrete as the incorporation ratios of PET flakes to replace CA increase at 7 days and 28 days. The results showed a consistent decrease in compressive strength with higher PET flakes content. Which was similar to the results obtained from the experiment conducted by Ismail Khalil and Jumaa Khalaf (2017). Unlike CA, PET flakes lacked interaction with the cement paste, resulting in a weaker interfacial transition zone (ITZ) in concrete, thereby reducing the compressive strength compared to CTR.

Moreover, in term of curing period, the percentage rising in compressive strength for CTR to PET-15 by following the arrangement in Figure 4.9 was increased from 7 days to 28 days. The compressive strength of CTR, PET-5, PET-10, PET-15 increased by 20.2 %, 20.9 %, 22 % and 27.5 % respectively from 7 days to 28 days as the hydration process in the early stage of concrete mixes with more PET flakes replacement was relatively inactive than CTR, which caused slower strength gain. However, the compressive strength of PET-15 was recorded as 24.07 MPa, which was not achieved the desired 25 MPa. Thus, the PET-15 was not suitable to be adopted for further experiment.

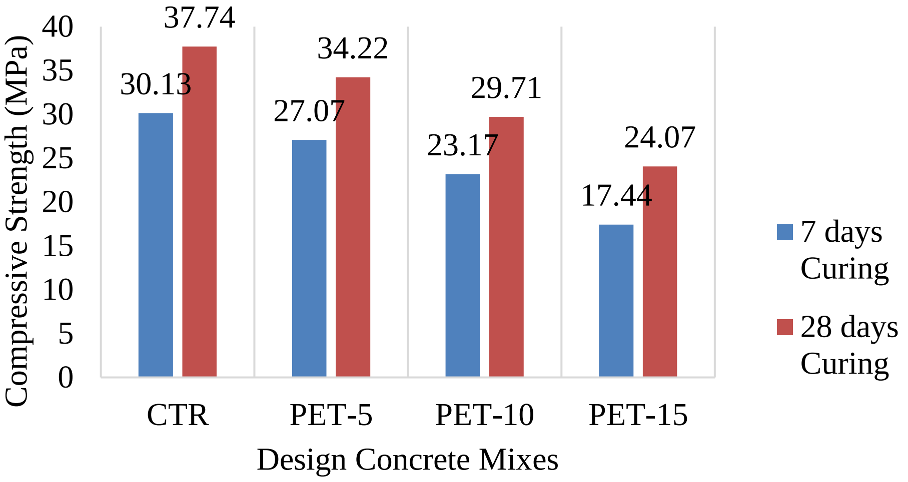


Figure 4.10: Compressive Strength of Various Design Concrete Mixes.

4.4.4 Splitting Tensile Strength

Based on the Figure 4.11 shows the splitting tensile strength of CTR, PET-5, PET-10 and PET-15 for 7 days and 28 days of curing ages. The results showed overall design concrete mixes exhibited an increase in splitting tensile strength as the curing time progressed. However, increased in PET flakes replacement in concrete mix has lowered the splitting tensile strength obviously for 7 days while insignificantly for 28 days. A similar explanation for the loss of compressive strength of concrete was applied to account for the behaviour of splitting tensile strength of the concrete mix with PET flakes replacement of CA due to weak interlock between cement paste and PET flakes.

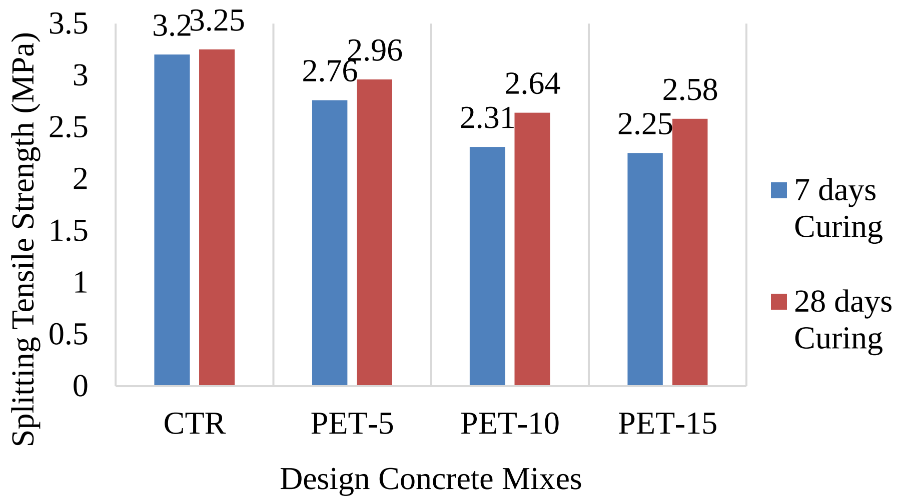


Figure 4.11: Splitting Tensile Strength of Various Design Concrete Mixes.

4.4.5 Summary

After analysing all the results, it was concluded that the slump values and overall density were decreased as the PET flakes content in concrete mix increased. Similarly, compressive and splitting tensile strength of the concrete were decreased with the increases of PET flakes content in concrete mix. Among all the design concrete mixes, PET-10 exhibited the most promising performance of the design concrete mixes, with a compressive strength of 29.71 MPa at 28 days, achieved the desired compressive strength of 25 MPa. PET-10 was also demonstrated greater sustainability compared to PET-5 as more waste PET flakes can be reused in concrete mix to meet the desired strength of 25 MPa, aligning with sustainable development objectives. Consequently, the optimum design mix proportions of PET-10 were adopted for further experimentation in Phase 3.

4.5 Actual Mix with MK Powder Replacement (Phase 3)

This chapter investigated the effect of substituting Orang Kuat Cement (OPC) powder with varying percentages of metakaolin (MK) in PET-10. Generally, MK has finer average particle sizes compared to OPC(Guignone et al., 2020). The experimental procedures encompassed assessments of fresh concrete properties, such as slump test and density determination, along with the hardened concrete test, including compressive strength and splitting tensile strength. Three design concrete mixes were prepared with partial replacement of unit weight of OPC by MK powder in different percentages ranging from 5 % to 10% in increments of 2.5 % replacing the unit weight of OPC. They were 5 % of MK replacing OPC (PET-10 + MK-5), followed by 7.5 % of MK replacement (PET-10 + MK-7.5) and 10 % of MK substitution (PET-10 + MK-10). The overall performances of design concrete mixes were concluded in summary and an optimum design concrete mix was selected. Table 4.7 computes the design mix proportions of PET-10 + MK-5, PET-10 + MK-7.5 and PET-10 + MK-10 respectively.

Table 4.7: Design Mix Proportions for 1 m3 of PET-10 + MK-5, PET-10 + MK-7.5 and PET-10 + MK-10.

Design Mix	**W/C Ratio**	**Unit Weight (kg/m^3)**					
		Binders		**Fine Aggregates (<4.75 mm)**	**Coarse Aggregates**		
		OPC	**MK Powder**		**10 mm Aggregates**	**20 mm Aggregates**	**PET Flakes**
PET-10 + MK-5	0.6	356.25	18.75	534.0	394.4	789.4	63.5
PET-10 + MK-7.5	0.6	349.72	25.28	534.0	394.4	789.4	63.5
PET-10 + MK-10	0.6	337.50	37.50	534.0	394.4	789.4	63.5

4.5.1 Fresh Properties

The fresh properties assessment included conducting slump test on PET-10 + MK-5, PET-10 + MK-7.5 and PET-10 + MK-10. As illustrated in Figure 4.12, reduction in slump value of approximately 20 % by increasing in MK powder contents with 2.5 % interval compared to PET-10. This indicated that the workability of the concrete mix is reduced when MK powder contents was increased. This is because the reduction in fineness modulus of cementitious materials led to lesser quantity of cement paste available to provide lubrication per unit surface area of the aggregates (Jagtap et al., 2008). Furthermore, the slump value of 50 mm of PET-10 + MK-10 was lesser than 60 mm as per requirement. Thus, the PET-10 + MK-10 was rejected to be adopted due to dry slump that might cause honey corn during concrete hardening.

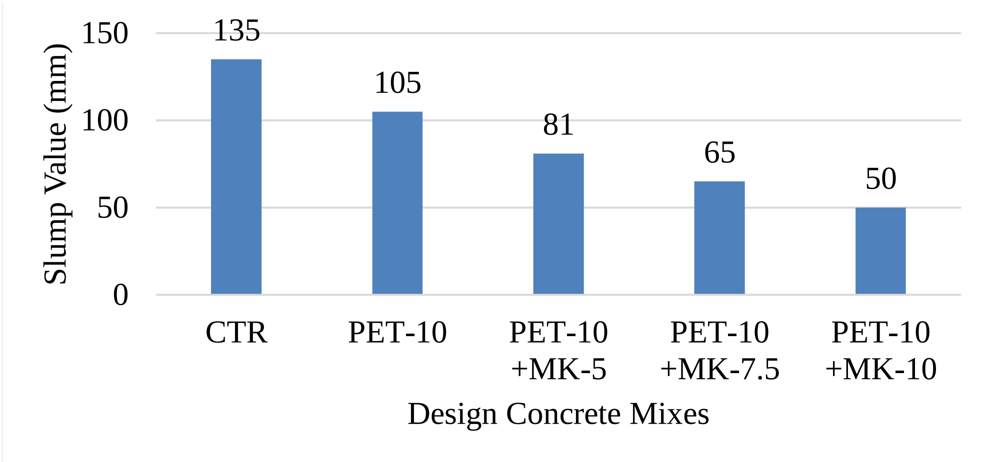


Figure 4.12: Slump Values of Various MK Content in PET-10.

4.5.2 Density

The fresh and hardened density of various MK content in PET-10 were shown in Figure 4.13. The fresh density of concrete mixes was greatly increased by 45 % when the 5 % of MK content while only 1 % ~ 2 % of fresh density were increased for 7.5 % and 10 % MK content. This is primarily due to the finer particle size and higher surface area of MK powder, which result in a denser mixture. On the other hand, hardened density of concrete mixes was insignificantly increased with increased in MK content as MK enhanced the packing of particles which to a more uniform compact microstructure.

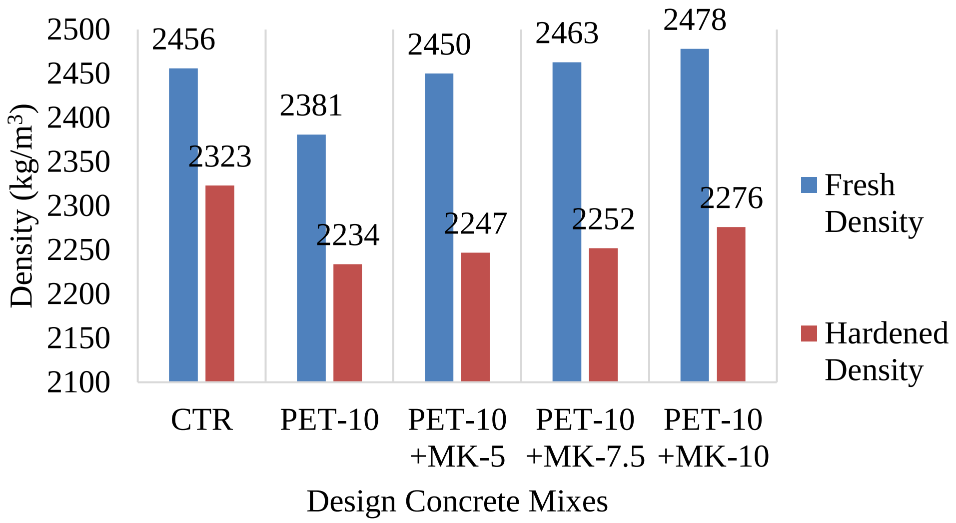


Figure 4.13: Fresh and Hardened Density of Various MK Content in PET-10.

4.5.3 Compressive Strength

Figure 4.14 demonstrates the compressive strength of concrete mixes for 7 days and 28 days were increased from PET-10 + MK-5 to PET-10 + MK-7.5 while decreased when the replacement was more than 7.5 % to PET-10. The significant increase in compressive strength with a peak value of 37.71 MPa at 28 days age of PET-10 + MK-7.5 has increased 27 % from PET-10, which was similar to the results obtained from CTR. The finding showed the similar outcome from the research carried out by (Angadi et al., 2018). MK improved the concrete compressive strength as the silica presented in MK was reacts with Calcium Hydroxide, Ca $(OH)_2$, a byproduct of hydration process of cement, which was forming additional calcium silicate hydrae (C-S-H) gel to gain the strength. This pozzolanic process enhanced strength until reaching a saturation point. Once the replacement exceeded 7.5 % MK replacement, workability of fresh concrete was too dry that led to increasing porosity and not enough calcium hydroxide for the reaction to complete. Therefore, the optimum percentage of MK replacement of OPC was 7.5 %, PET-10 + MK-7.5.

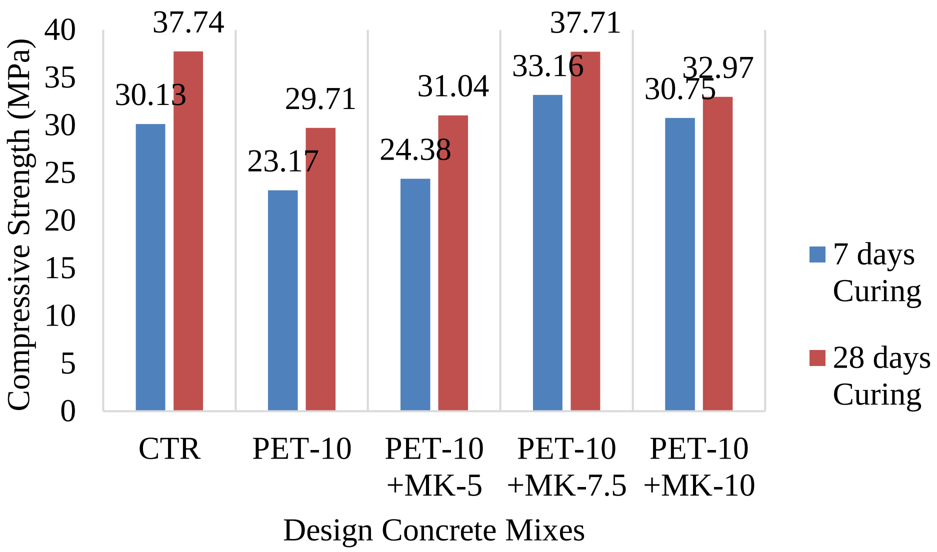


Figure 4.14: Compressive Strength of Various MK Content in PET-10.

4.5.4 Splitting Tensile Strength

Figure 4.15 illustrates the splitting tensile strength of CTR, PET-10, PET-10 + MK-5, PET-10 + MK-7.5 and PET-10 + MK-10 for 7 days and 28 days of curing ages. The results revealed an insignificant increased in splitting tensile strength of PET-10 + MK-5 and greatly increased by 14.39 % of PET-10 + MK-7.5 with the peak value of 3.02 MPa at 28 days age compared to PET-10. Nevertheless, PET-10 + MK-10 exhibited decreased in splitting tensile strength of 2.9 MPa at 28 days age as showed similar to the result trends of Figure 4.14. Thus, PET-10 + MK-7.5 provided the optimum splitting tensile strength.

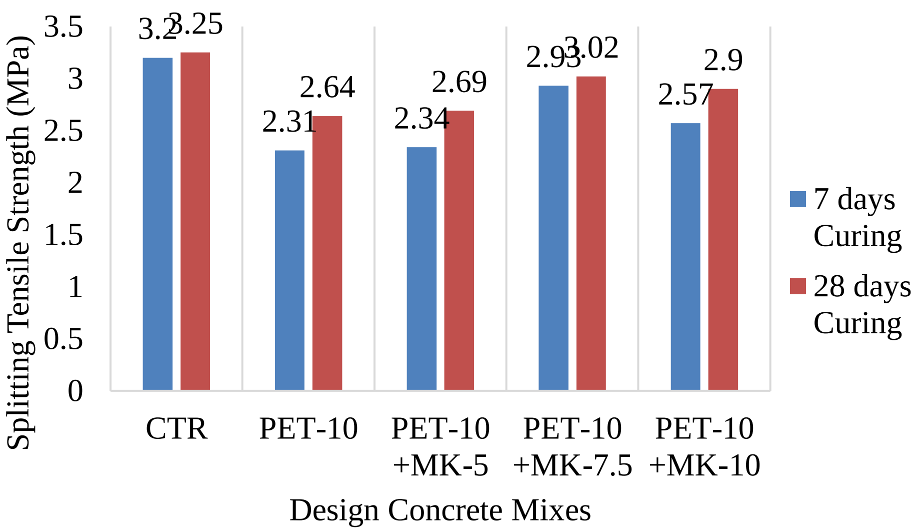


Figure 4.15: Splitting Tensile Strength of Various Design Concrete Mixes.

4.5.5 Summary

Upon thorough analysis of the findings, it was determined that the slump values decreased with an increased in MK powder content. Oppositely, fresh and hardened density increased with increased in MK powder content. In term of concrete strength, increased in MK powder content has improved the compressive and splitting tensile strength, while dropped when the MK powder content was exceeded 7.5 %. Notably, among all the designed concrete mixes, PET-10 + MK-7.5 showed the most promising performance, boasting a peak compressive strength of 37.71 MPa at 28 days, which was achieved the desired 25 MPa. Therefore, the optimal design mix proportion of PET-10 + MK-7.5 was selected.

4.6 SEM - EDX Analysis

Scanning electron microscopy (SEM) was applied in this project to acquire a microstructural and chemical characterization of design concrete specimens. Where the Energy Dispersive X-Ray Analyser (EDX) was an add-on Electron Microscopy tools for elemental identification and quantitative compositional data. Figure 4.16 and Figure 4.17 shows the SEM and EDX analysis of Metakaolin (MK). Figure 4.16 illustrates the SEM Morphology of MK at magnification of 9000 and MK exhibited a flake-like morphology and tends to aggregate or stack together.

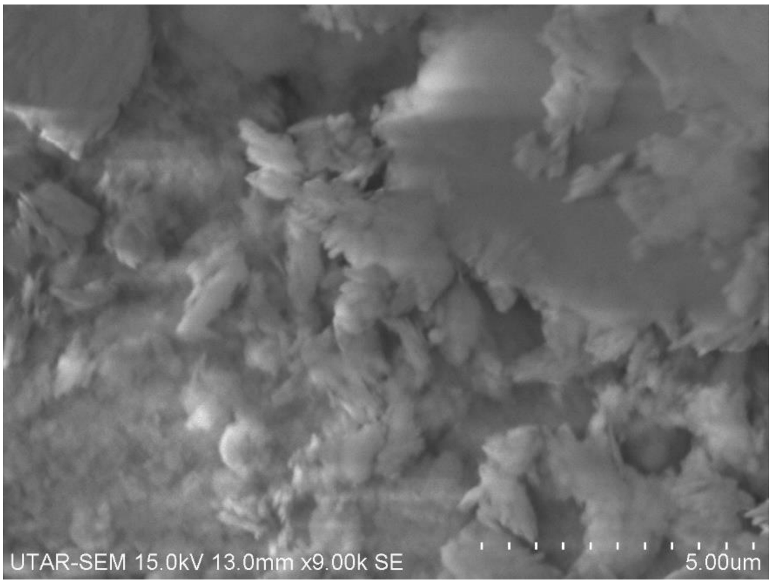
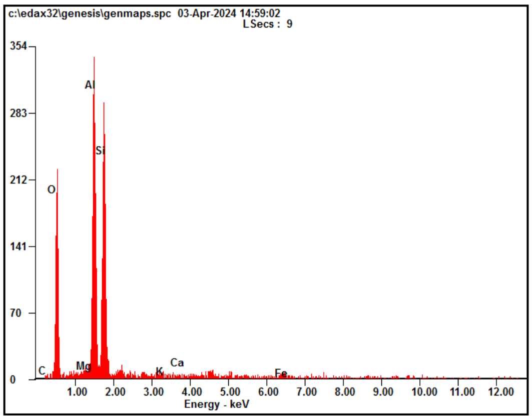


Figure 4.16: SEM Morphology of MK.

Moreover, Figure 4.17 shows the elemental composition with weight and atomic weightage of MK. The finding revealed that MK contained high content of silicon (Si) and aluminium (Al) elements with the amount of 31.25 % of weight weightage and 23.71 % of atomic weightage for Si, whereas 25.67 % of weight weightage and 20.28 % of atomic weightage for Al. Si and Al oxides were the main composition that improved the strength of the concrete.



Element	*Wt%*	*At%*
CK	01.96	03.47
OK	38.46	51.24
MgK	00.27	00.24
AlK	25.67	20.28
SiK	31.25	23.71
KK	00.47	00.26
CaK	00.41	00.22
FeK	01.51	00.58
Matrix	Correction	ZAF

Figure 4.17: Element Composition of MK.

Furthermore, Figure 4.18 illustrates that the SEM morphology of CTR under 6500 magnifications with detail labelled of the microstructure occurred. Calcium Silicate Hydrate (C-S-H gels) was the main hydration product that enhanced the concrete strength and cohesiveness. Calcium Hydroxide (CH) was formed mainly from alite (C_3S) hydration. Lastly, ettringite exhibited a rod-like crystals in the early stages of cement hydration with the chemical formula of $[Ca_3Al(OH)_6 \cdot 12H_2O]_2 \cdot 2H_2O$.

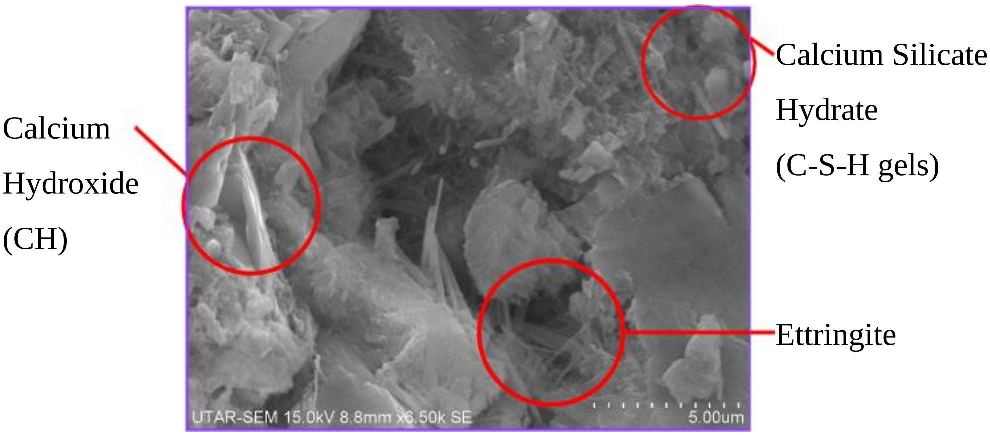
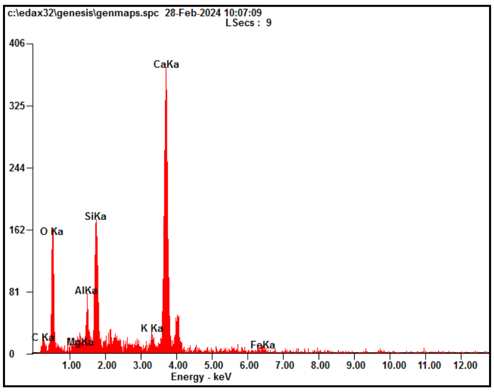


Figure 4.18: SEM Morphology of CTR.

Based on the Figure 4.19 reveals that the elemental composition with weight and atomic weightage of CTR. The finding exhibited that CTR has significantly high content of Calcium (Ca) with the amount of 35.68 % of weight weightage and 19.97 % of atomic weightage, whereas the second highest was Silicon with 10.68 % of weight weightage and 8.53 % of atomic weightage. Typically, the Ca/Si ratio of the concrete was 0.8 to 2.5. However, the Ca/Si ratio of CTR was 2.34 which was within the acceptable range of Standard Ca/Si ratio range.



Element	*Wt%*	*At%*
CK	06.20	11.57
OK	38.21	53.56
MgK	01.26	01.16
AlK	03.93	03.26
SiK	10.68	08.53
KK	01.85	01.06
CaK	35.68	19.97
FeK	02.19	00.88
Matrix	Correction	ZAF

Figure 4.19: Element Composition of CTR.

Lastly, SEM and EDX analysis of the optimal design concrete mix of PET-10 + MK-7.5 was conducted to understand the microstructure of the concrete and make a comparison with CTR. Figure 4.20 illustrates the SEM morphology of PET-10 + MK-7.5 under 90 of magnification with the large area of C-S-H gels, a large particle of PET flakes and void gap in between cement paste and PET flakes due to weak interlock of PET to cement paste.

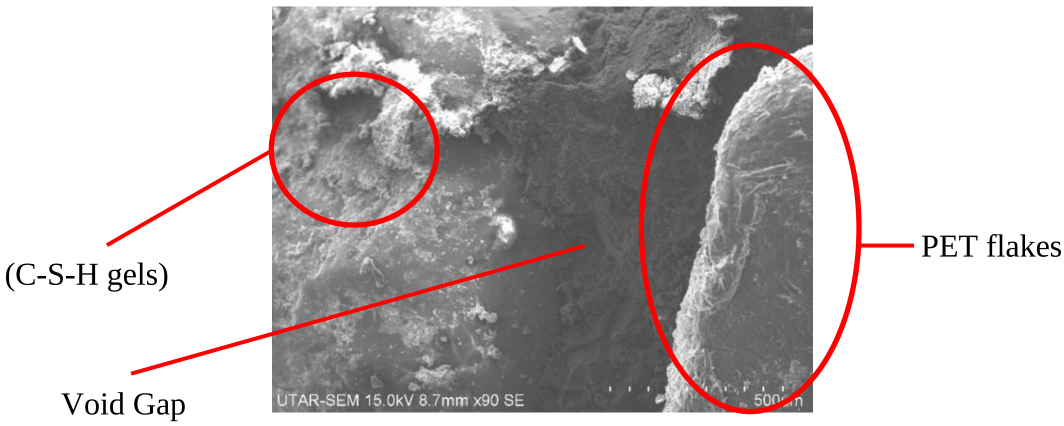
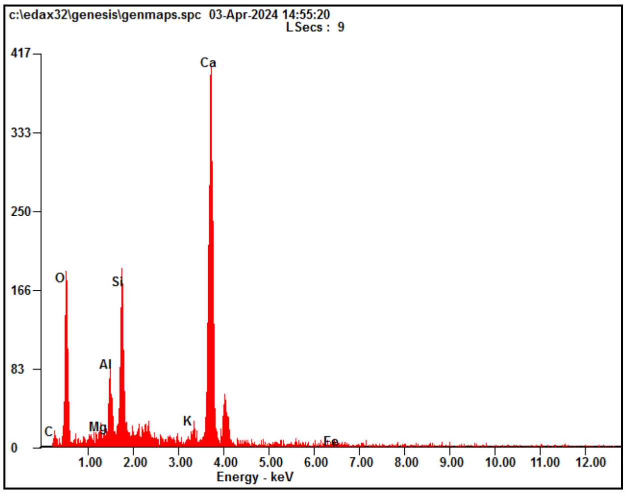


Figure 4.20: SEM Morphology of PET-10 + MK-7.5.

Figure 4.21 demonstrates the elemental composition with weight and atomic weightage of PET-10 + MK-7.5. The findings revealed that PET-10 + MK-7.5 also exhibited a significantly high content of Calcium (Ca). Silicon (Si) emerged as the second highest content, constituting 10.74 % of weight weightage and 8.55 % of atomic weightage. The Ca/Si ratio of PET-10 + MK was 2.28, which was slightly lower than CTR with 2.34. The lower the Ca/Si ratio indicated that more polymerized or denser C-S-H gels formed. Therefore, addition of MK in PET-10 enhanced the strength of the concrete from 29.71 MPa to 37.71 MPa in order to achieved similar compressive strength of CTR.



***Element*¤**	***Wt%*¤**	***At%*¤**
··***CK***¤	03.63¤	06.75¤
··***OK***¤	42.02¤	58.71¤
·***MgK***¤	01.50¤	01.38¤
·***AlK***¤	04.74¤	03.93¤
·***SiK***¤	10.74¤	08.55¤
··***KK***¤	01.23¤	00.70¤
·***CaK***¤	35.02¤	19.53¤
·***FeK***¤	01.13¤	00.45¤
Matrix¤	Correction¤	ZAF¤

Figure 4.21: Element Composition of PET-10 + MK-7.5.

4.7 XRD Analysis

X-Ray Diffraction (XRD) analysis was performed to analyse the crystal structure of the concrete specimens by assessing the diffraction pattern produced when X-rays interacted with the atomic lattice of a crystalline material such as cementitious materials. Based on the Figure 4.22 shows the intensity (counts) against the 2θ / Degrees of MK. The results exhibited peaks percentage of amorphous silica (SiO_2) and low intensity of calcium carbonate ($CaCO_3$). According to the Figure 4.22, MK contained more quartz phase and also Aluminium (Al) chemical composition, thus, it was supposed to have the high intensity of chemical compound of Al_2O_3 as discussed in the research conducted by Dai et al. (2022). This indicated the presence of great numbers of amorphous silicoaluminate minerals were highly reactive mineral.

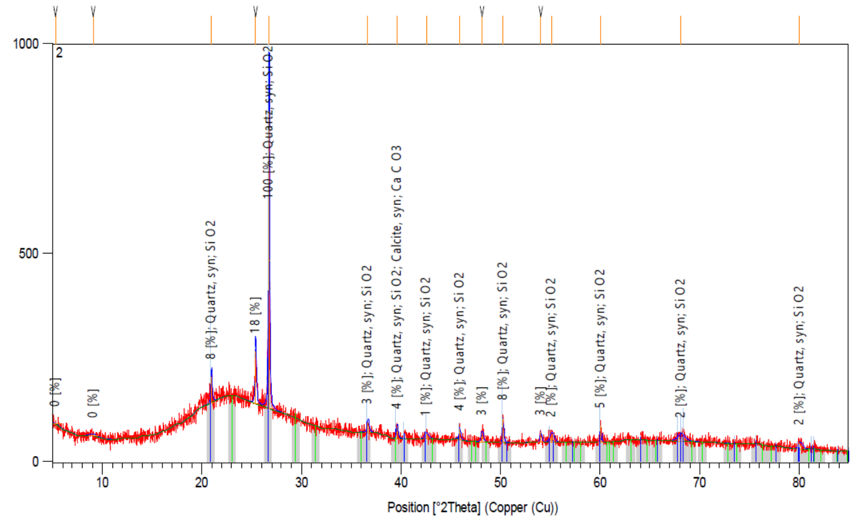


Figure 4.22: XRD Results of MK.

In the Figure 4.23 and Figure 4.24 illustrates the intensity (counts) against the 2θ / Degrees of CTR and PET-10 + MK-7.5 specimens respectively. The finding of Figure 4.23 and 4.24 reveals the similar peak intensity of Quantz at degree of 26. Where Figure 4.23 shows the low intensity of Calcite and Iron (III) oxide (Fe_2O_3), Hematite. On the other hand, Figure 4.24 indicates that PET-10 + MK-7.5 contained Copper Iron Sulphide (Cu_5FeS_4), Bornite and Calcium Fluoride (CaF_2), Fluorite. However, there were several chemical compounds that not listed in the figure but was existed in concrete, namely Calcium Silicate Hydrate (C-S-H gels), Calcium Hydroxide (CH) and Ettringite. C-S-H gels was the main hydration product that gain the concrete strength where the CH was product from alite hydration that the excess lime is available.

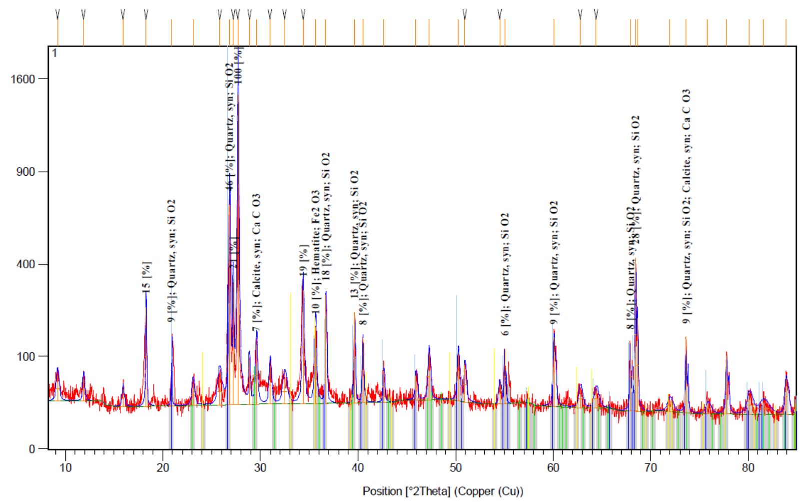


Figure 4.23: XRD Results of CTR.

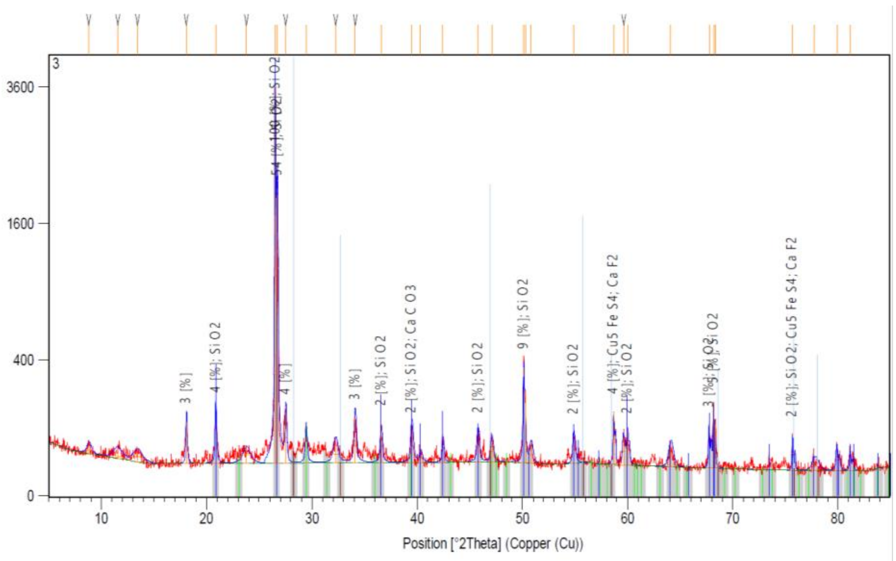


Figure 4.24: XRD Results of PET-10 + MK-7.5.

4.8 Summary

In conclusion, trial mix of CTR and PET-5 were carried out in Phase 1 to obtain the optimal W/C ratio of the concrete mix that was achieved the desired compressive strength of 25 MPa. In Phase 2, increases in replacement of CA with PET flakes reduced the workability and density of the concrete as well as the compressive and splitting tensile strength of the concrete due to weak interlock between the cement paste and PET flakes. Based on the overall performance of the designed concrete, design mix proportion of PET-10 was selected for further experimental work in Phase 3. In Phase 3, the finding exhibited that MK powder replacement of OPC was reduced the workability but slightly increased in overall concrete density. In term of hardened properties, MK replacing OPC improved the concrete compressive strength and splitting tensile strength. However, the strengths were dropped when the MK substitution was exceeded 7.5 % as the water demand increased caused more void and porosity occurred. It is also because of not sufficient CH to complete the reaction to form CSH. Therefore, PET-10 + MK-7.5 was selected to be optimal design concrete mix as it provided the greatest overall performance. Figure 4.25 demonstrates all of the compressive strength of the design concrete mixes, where the PET-10 + MK-7.5 exhibited the greatest compressive strength.

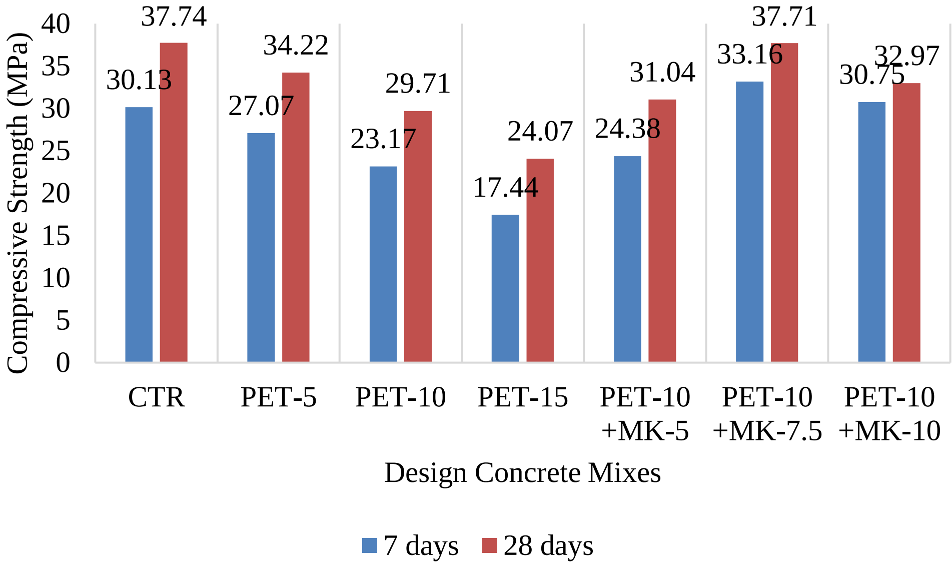


Figure 4.25: Summary of Compressive Strength of Design Concrete Mixes.

Additionally, the microstructural analysis of SEM- EDX and XRD analyses were performed to acquire a microstructural and chemical characterization of design concrete specimens. MK contained high weight and atomic weightage of Si and Al, which provided the pozzolanic reaction that was increased the overall concrete strength. Where the concrete contained C-S-H gels that was mainly for strength enhancement as well as CH.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

After a series of thorough experiments were carried out, conclusions were drawn related to the objectives outlined at the beginning of this study.

The first objective of this study is to design a concrete mix proportion with grade of 25 MPa. This objective was achieved by conducting the trial mixes of CTR and PET-5 with varying W/C ratio (Phase 1). W/C ratio of 0.6 was selected for further fresh and hardened concrete experiment compared to 0.65 and 0.7 as 0.6 W/C ratio for both design mixes met the desired compressive strength of 25 MPa at 28 days.

The second objective is to investigate the optimal percentage between 5 %, 10 % and 15 % replacement of PET for coarse aggregate and effect to achieve concrete grade 25 MPa compressive strength. This objective was achieved during the Phase 2. Three sets of design concrete mixes with various amount of PET flakes from 5 % to 15 % with an interval of 5 % were prepared. Several experimental tests were conducted to determine the effect of PET flakes replacing CA on various concrete properties. These tests included slump test, fresh and hardened density, compressive and splitting tensile strength. Based on sustainability consideration and achieving requiring strength of 25 MPa, PET-10 was selected as optimal design concrete mix for further experimental works in Phrase 3.

The last objective is to synthesize the effect of metakaolin replacement for OPC under 5 %, 7.5 % and 10 %. This objective was fulfilled during Phase 3. Three sets of design concrete mixes were casted with different percentage of MK from 5 % to 10 % with an 2.5 % interval. The concrete properties tests that included in this study in order to assess the effect of MK replacing OPC were slump test, fresh and hardened density, compressive and splitting tensile strength. Therefore, PET-10 + MK-7.5 was selected as the optimal design concrete mix.

5.2 Recommendations

Several recommendations could be considered to enhance validation and achieve more reliable results for future studies:

(i) Study the effect of PET flakes with varying shapes and sizes (PET powder, PET fibre and PET fine aggregate) on the concrete properties to optimise the research study.

(ii) Study on the different types of sustainable materials such as food waste, agricultural waste or construction waste can be used in the concrete mix design to achieve sustainable development goals.

(iii) Study the other concrete properties such as compaction factor test and impact factor test of the designed concrete to optimise the research study.

(iv) Study the long-term behaviour of designed concrete that is beyond 28 days of curing such as 56 days and 90 days to investigate the pozzolanic effects of MK in designed concrete, durability and the changes in mechanical strengths after long periods of curing.

(v) Study the mechanical properties and durability of designed concrete under different environmental condition. For instead, exposure to the seawater, acidic environment or critical environment.

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