PERFORMANCE EVALUATION OF GEOPOLYMER MORTAR WITH SUSTAINABLE PALM OIL CLINKER SAND USING A VOLUME BASED APPROACH

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PERFORMANCE EVALUATION OF GEOPOLYMER MORTAR WITH SUSTAINABLE PALM OIL CLINKER SAND USING A VOLUME BASED APPROACH

ABSTRACT

Excessive sand exploitation is believed to be one of the main factors for lowering groundwater and flooding, resulting in erosion. This research focused on replacing the conventional sand with a local industrial by-product, palm oil clinker (POC), to incorporate in mortar and concrete. The physical properties, chemical composition, and microscopic images of POC sand and conventional sand were compared. Due to POC's porous nature and irregular shape, a volume-based approach was applied for replacements and to design mixes based on ASTM codes of practice. Fresh and hardened characteristics such as mechanical properties, durability, and microstructural investigations were performed in order to study the effect of POC in cement and geopolymer mortars and concrete. In addition, the effect of different gradings of POC sand was studied in both cases of cement and geopolymer mortars to have a better understanding of this by-product. To promote the sustainability of the geopolymer products, the mixes were cured at ambient condition to eliminate the unnecessary oven-curing method. This was possible with the incorporation of ground granulated blast-furnace slag (GGBS), which is a by-product of the iron mill. POC sand showed a comparable result in cement and geopolymer mortars to those of mining sand mortars. However, a slight decrease in the compressive strength was reported while benefiting from a lower density. It was found that the use of finer particles of POCS decreased the flowability while it increased the compressive strength. Furthermore, the sodium silicate present in the geopolymer was replaced with an identified batik waste generated from the batik factories with different percentages. With up to 60% replacement of commercial sodium silicate with batik waste, the 28-day compressive strength experienced a negligible reduction of less than 5% to achieve 65

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MPa. The utilization of batik waste as the replacement for conventional commercial silicate demonstrated acceptable performance in geopolymer mortar and concrete. The incorporation of these waste materials in concrete could improve sustainability, reduce the cost, and mitigate the environmental impacts. To address the application of the findings, the materials and the concept were further deployed in concrete and the production of hollow blocks.

Keywords: geopolymer mortar, palm oil clinker, sodium silicate, sand replacement, sustainability

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PENILAIAN PRESTASI GEOPOLYMER MORTAR DENGAN KLINKER MINYAK SAWIT YANG LESTARI DENGAN MENGGUNAKAN PENDEKATAN BERASASKAN VOLUME

ABSTRAK

Eksploitasi pasir secara berlebihan dipercayai merupakan faktor utama penurunan air tanah dan banjir yang mengakibatkan hakisan. Kajian ini memberi tumpuan kepada penggantian pasir konvensional dengan produk sampingan dari industri tempatan, klinker minyak sawit (POC), untuk digunakan dalam mortar dan konkrit. Ciri-ciri seperti sifat fizikal, komponen kimia, dan mikrostruktur antara POC dan pasir konvensional dibandingkan. Oleh kerana sifat berliang POC dan bentuk yang tidak teratur, pendekatan berasaskan isipadu digunakan untuk penggantian dan merangka campuran berdasarkan peraturan ASTM. Ciri-ciri baru dan keras seperti sifat-sifat mekanikal, kajian ketahanan dan penyiasatan mikrostruktur telah dilakukan untuk memerhatikan kelakuan POC dalam simen dan mortar dan konkrit geopolimer. Di samping itu, kesan daripada gradasi POC yang berbeza telah dipelajari dalam kedua-dua kes simen dan mortar geopolimer untuk mempunyai pemahaman yang lebih dalam mengenai produk sampingan ini. Untuk menggalakkan kelestarian produk geopolimer, campuran telah diawet dalam keadaan ambien untuk mengelak daripada kaedah pengawetan ketuhar. Ini adalah berkemungkinan dengan penggabungan sanga tanah granulated (GGBS) yang merupakan produk sampingan dari kilang besi. POC menunjukkan prestasi yang boleh dibandingkan dengan mortar simen dan geopolimer dengan mortar pasir perlombongan yang menunjukkan sedikit pengurangan dalam ujian mampatan walau bagaimanapun mempunyai kepentingan dalam menghasilkan ketumpatan yang rendah. Ia didapati bahawa penggunaan saiz partikel POC yang halus mengurangkan kadar kebolehaliran dan kekuatan lenturan mortar tetapi meningkatkan kekuatan mampatan. Tambahan lagi, sodium silikat yang terdapat dalam geopolimer telah digantikan dengan sisa air yang telah

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dikenal-pasti dan dihasilkan dari kilang-kilang batik dengan peratusan yang berlainan. Dengan penggantian natrium silikat komersil hingga 60% dengan air sisa batik, kekuatan mampatan 28 hari mengalami pengurangan yang boleh diabaikan iaitu kurang daripada 5% untuk mencapai 65 MPa. Pasir POC dan air sisa batik menunjukkan hasil penggabungan yang sangat baik di dalam mortar geopolimer dan konkrit. Hasil gabungan daripada bahan sampingan di dalam konkrit boleh meningkatkan kelestarian, mengurangkan kos, dan mengurangkan kesan kepada persekitaran. Untuk mempraktikkan hasil kajian yang diperoleh, bahan dan konsep tersebut terus digunakan dalam konkrit dan penghasilan blok berongga.

Kata kunci: mortar geopolimer, klinker minyak sawit, natrium silikat, penggantian pasir, kelestarian

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LIST OF SYMBOLS AND ABBREVIATIONS

AAS	:	Alkaline activator solution
ASTM	:	American society for testing and materials
EDX	:	Energy Dispersive X-Ray Analyzer
FA	:	Fly ash
GGBS	:	Ground granulated blast-furnace slag
MS	:	Mining sand
OPS	:	Oil palm shell
POC	:	Palm oil clinker
POCS	:	Palm oil clinker sand
POFA	:	Palm oil fuel ash
SEM	:	Scanning Electron Microscope
UPV	:	Ultrasonic pulse velocity
XRD	:	X-ray Diffraction
XRF	:	X-ray diffraction

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CHAPTER 1: INTRODUCTION

1.1 Background

Mortars have been an inseparable part of the infrastructure in human lives ever since they were introduced right from the very first mortars containing mud and clay to the modern mortars of cement paste and fine aggregates. Strength and durability, along with the capability of incorporating organic and inorganic local materials, have made mortars effortlessly achievable and trustworthy in addition to having the privilege of relatively inexpensive components. Mortar is one of the first products to utilize recycled substances to enhance the integrity of its performance.

During the past few decades, the depletion of huge quantities of natural resources alarmed many developed, developing, and under-developed nations to take drastic steps to impose restrictions on the usage of conventional materials. Due to the development and expansion of the infrastructures and the ever-increasing need for the housing and the industry, more raw materials are being exploited. On the contrary, more waste materials are being generated by many industries and some of those materials underutilized and mostly dumped in the landfills.

Recycling and reusing the waste materials could be the most reasonable act in tackling both issues of preservation of the natural resources and the proper handling of the waste materials as it contributes to the sustainability and preservation of virgin materials. In major developing economies, such as India, China, and other Southeast Asian countries, millions of tons of industrial by-products, such as fly ash, ground granulated blast furnace slag, quarry dust, and steel slag, are considered to be potential replacements for the conventional construction materials.

One of the most vital natural resources that has been overlooked is sand. The formation of sand takes thousands of years of eroding and breaking down of the rocks while it is consumed in a reasonably short period. Sand could directly affect the main properties of

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the final product and prevent the shrinkage of the final mortar. Nevertheless, the over usage of this natural fine aggregate has led to a substantial shortage in specific areas such as Southeast Asia. Thus, many researchers have focused on replacing sand with more sustainable materials, which largely includes the utilization of waste materials. Considering the disposal issue that is caused by large amounts of industrial wastes generated by factories, the utilization of by-products could mitigate the environmental pollution.

Palm oil industry is one of the largest industries in Malaysia that generates large amounts of waste from which palm oil clinker (POC), a by-product generated during the processes, has gained the attention of researchers as a replacement of fine and coarse aggregates in the concrete. Nevertheless, more in-depth study on the methodology behind the utilization of this by-product is needed in order to optimise the performance of the products utilizing POC.

A volume-based method has been proposed in this study for the replacement of conventional sand with POC. This replacement method uses the specific gravity of the materials rather than the weight-based replacement used in the literature. The efficiency of this replacement has been studied in the cement mortar utilizing POC; the calculation along with the experimental work have been compared.

Moreover, the emergence of the geopolymer concrete (cement-free concrete) has opened new possibilities of utilizing waste materials as aggregates and binders in the construction industry. Geopolymers are formed by a chemical reaction that is different to that of conventional concrete. Thus, the present study has emphasized on the usage of POC as a replacement of conventional sand in the geopolymer mortar as well as the conventional mortar.

Nevertheless, due to the higher cost of geopolymers compared to the conventional concrete, the geopolymer concrete has not been accepted as a replacement of the

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conventional concrete. In order to lower the cost of the geopolymer, a waste material from batik industry has been introduced to be used as the silicate source in the mixes.

1.2 Problem statement

The poriferous nature of the POC has been an obstacle in the utilization process of it in the concrete. In the literature, the replacements of the fine and coarse aggregates with POC were performed based on the weight, and the issue about this methodology is that the same weight of the POC to that of conventional aggregates, will have much higher volume. The concept of the sand is to act as a filler in the concrete to reduce the cost of the mix, to increase the volume of the concrete, and more importantly to contribute to the strength and reduction of the shrinkage.

Considering these facts, the volume of the sand plays a vital role in determining the overall performance of the final product. This research employs a volume-based approach to utilize POC in the geopolymer mortar and concrete. The volume-based design facilitates the replacement process of the sand since it considers the inner voids of the particles. The utilization of POC in mortar and concrete could pave the way for broad usage of this by-product. The study of behaviour of POC in geopolymer is very limited at the time of conducting this study and thus, the focus of this research has been on the geopolymer mortar containing palm oil clinker as the sole fine aggregate.

This research also focused on the replacement of the sodium silicate used as the activator in the geopolymerization process along with the sodium hydroxide. The manufacture of sodium silicate causes huge CO_2 emissions along with its high cost which could question the whole idea of using geopolymer concrete over conventional concrete because of CO_2 emissions of cement. A batik waste from the batik industry was identified as a potential sodium silicate source to be used as the alkaline activator.

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1.3 Research objectives

The focus of the current research was investigating POC as an alternative to the sand as well as to develop sustainable POC based geopolymer mortar.

1. To investigate the characteristics of POCS and to develop cement mortar with the replacement of conventional sand with POCS using a volume-based approach
2. To examine the mechanical, microstructure, and durability properties of POCS in geopolymer mortar
3. To study the effect of different gradings of POCS as sand replacement on the properties of cement and geopolymer mortars
4. To investigate the feasibility of utilizing the batik industry batik waste as silicate source in geopolymer mortar
5. To appraise the performance of the mechanical properties of geopolymer concrete and hollow blocks with the incorporation of POC as fine and coarse aggregates as application

1.4 Research significance

The main advantages of the current research could be categorized in the following two stages.

1. The replacement of the POC instead of conventional sand could bring about numerous benefits. The foremost benefit is the preservation of the natural resources as some of the regions have already facing conflicts over the shortage of the sand and the opportunity of having an alternative sand with a comparative performance to the conventional one could alleviate the complications. Furthermore, POC is a lightweight material which could significantly reduce the overall weight of the structure.

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2. The batik waste from the batik industry contains chemicals and heavy metals and disposing it may cause harm to the environment. Incorporating batik waste in the geopolymer activation process could be a solution to the disposal issue and moreover a reduction in usage of commercial sodium silicate.

1.5 Scope of research

In the first stage, prior to the utilization of POC in the mixes, its material properties were tested and identified since it is an important step in defining the behaviour of the material. Physical properties, along with the microstructural analyses, were performed on the POC and other materials used in this research in advance to the trial mixes and the preparation of the mix designs.

In the second stage, the performance of the POC in cement mortar was evaluated, and a comprehensive study on developing a volume-based mix design for POC cement mortar was performed. Afterwards, through the third stage, geopolymer mortar was developed with the incorporation of POC. Different aspects of the acquired samples were analysed in terms of mechanical performance, durability studies, and in-depth microstructural investigations.

Afterwards, the effect of grading of the palm oil clinker sand was evaluated in conventional and geopolymer mortars and then the batik waste's replacement of the commercial sodium silicate was studied in terms of mechanical, microstructure, and chemical resistance.

Lastly, the findings of this research were used to analyse a POC-based geopolymer concrete and geopolymer hollow block cured in the ambient condition as an application to the findings of the current study.

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1.6 Thesis organisation

Chapter 1 presents the problem statement and research objectives along with the research significance and scope of research. Chapter 2 highlights a comprehensive study of the literature on the palm oil industry wastes, palm oil clinker, and geopolymer. In Chapter 3, the methodologies, materials, characterization of POCS, and the experimental test procedures are discussed. Chapter 4 presents the findings of the utilization of POCS in cement and geopolymer mortars, along with the effect of POCS particle size on the engineering properties of the mixes. In chapter 5, the feasibility of the incorporation of batik waste in geopolymer mortar is discussed and geopolymer concrete and hollow blocks using POCS and batik waste are highlighted as applications to the findings of the current study. Lastly, Chapter 6 presents a summary of the outcome of the thesis.

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CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

The fast-growing civilization has led to a greater egoistic culture through excellence in the infrastructure; thus, the need for construction of giant projects and networks of transportation is ever-growing. One of the most important and well-known materials in the construction sector is concrete worldwide. Its unique and versatile features on one hand and the relatively inexpensive production, on the other hand, has made it an appealing building material. However, during the production phase of cement, the main ingredient of concrete, huge amounts of CO_2 is released into the atmosphere. It is well known that the cement industry is responsible for about 5-8% of annual greenhouse gas emissions globally (Krausmann et al., 2009). These greenhouse gases are one of the main reasons of the global warming effect for not letting the heat depart from the atmosphere.

Consuming less energy, using substitutes to cement and minimizing the use of natural resources could pave the way to lessen the amount of CO_2 generated in the construction sector (Suhendro, 2014). The importance of this matter could be supported by a study (Li et al., 2019b) revealing that steel and cement are the two main culprits of 90% of total greenhouse gas emissions in reinforced-concrete structures.

On the other hand, the thirst for construction materials by the developing countries such as China, and India along with Middle Eastern countries for high rise and other dwellings, resulted in substantial exploitation of virgin materials. The consequence of such exploitation has caused much misery to habitats as well as an ecological imbalance. Thus, many developed and developing countries emphasize sustainable development in line with the United Nations' sustainable development goals (SDG) (United Nations, 2018). In order to fulfil the SDGs, many researchers have focused on utilizing industrial by-products, recycling materials and incorporating environmentally friendly materials. In

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concrete, the role of fine and coarse aggregates cannot be underestimated as they occupy up to about 60-80% of the volume.

The volume of solid waste materials generated is increasing worldwide. These wastes could be generated from thermal power plants, iron and steel industries, wood and cement manufacturers and disposal of these generated wastes need a huge area, operation cost, and other resources. The shortage of natural resources on the one hand and the issue of tackling huge waste materials, on the other hand, have made an opportunity to reuse the waste materials and further utilize them in the construction sector.

2.2 Sand scarcity

Cement and water that form the paste have been at the centre of attention for decades; the focus was mainly on finding potential alternatives for cement such as natural pozzolans, industrial by-products, etc. Although more attention had been given on replacing cement due to global warming, the role of other building materials such as sand and granite, should not be overlooked as they directly affect the strength, workability, drying shrinkage and appearance of the final product.

Sand is like a reservoir that many sectors such as construction, asphalt, glass manufacture, etc. depend on. Although the process of formation of sand takes a longer period, it is consumed in a very short period due to the high demand for construction that is mainly attributed to the population growth. It is becoming a scarce resource (Milliman & Syvitski, 1992), causing major issues for the construction industry and also a concern for the environment due to its overuse. Consumption of sand worldwide has exceeded 40 billion tons annually, which is twice the annual sediment of all rivers globally (Milliman & Syvitski, 1992). Sand extraction and exploitation affected the ecosystem that resulted in erosion, loss of species and ecological damages such as floods and tsunami magnification (Smith, 2018; Sutherland et al., 2017). Sand scarcity is much severe in Asia

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and Southeast Asia due to many mega projects and ever-increasing construction. Singapore is the greatest importer and consumer of sand per inhabitant (United Nations Environment Programme, 2014).

Although sand acts as a filler in concrete, it is a vital component as it increases the volume of concrete and reduces the shrinkage and the overall cost of the product. The shape, size, rigidity, porosity, etc. are some of the main factors that define the performance of the sand in concrete. Depending on the type of the sand, whether they are naturally formed by erosion or they are formed by crushing bigger flakes, the surface of the particles could be rough or smooth. Moreover, having impurity in sand is another factor that could adversely affect the quality of the product. Finding alternative sand for construction purposes demands a great deal of research in order to fulfil all the required properties.

Replacing the sand with industrial wastes and by-products could be an essential step towards developing sustainable construction materials (Bashar et al., 2016; Kanadasan et al., 2018). The need for natural aggregates has surpassed the availability in some countries, and thus, their dependency on the import section for satisfying their demands is indisputable (Thomas et al., 2018). This signifies the need for adopting greener construction materials and technologies.

2.3 Industrial wastes

With the advent of more industries in many developed and developing countries, the generation of waste and by-products is increasing, which could lead to a greater processing need for handling and dumping these wastes. In major developing economies such as India, China, and other Southeast Asian countries, millions of tons of industrial by-products such as fly ash, ground granulated blast furnace slag, quarry dust, steel slag, etc. are observed as a potential replacement for construction materials. One of the major

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industries in South East Asia is the palm oil industry which produces large amounts of palm wastes and approximately 85% of the global palm oil production belongs to Indonesia and Malaysia. Nevertheless, the amount of generated solid mass by the palm oil industry in Malaysia is reported to be 80 million tonnes (Samiran et al., 2016). Malaysia could greatly benefit from the incorporation of the wastes in concrete derivatives as one of the major exporters of palm oil, the environmental impact of managing the wastes could be alleviated.

In the recent years, the palm oil industry has shown the potential of producing large amounts of biomasses as reusable sources such as palm oil fuel ash (POFA), palm oil clinker (POC), oil palm shell (OPS), empty fruit bunches (EFB) and oil palm trunks (OPT) (Loh, 2017; Shafigh et al., 2013). Thus, a considerable amount of wastes from the palm oil industry requires proper handling. Studies have been conducted on the performance of palm oil by-products as a replacement of binder, sand and coarse aggregate in the cementitious products (Ahmmad et al., 2017; Alengaram et al., 2016; Mo et al., 2015; Nayaka et al., 2018). Incorporating OPS as a replacement of coarse aggregate in concrete, demonstrated its resistivity against impact and blast loads (Alengaram et al., 2016). In another study (Jagarapu & Eluru, 2019), researchers used OPS to reduce the density of the concrete and reported that the recommended replacement level is up to 30% with GGBS and POFA as binders. It has also been reported (Ul Islam et al., 2016) that OPS concrete produced comparable compressive strength and better durability with the whole replacement of conventional crushed granite aggregate with OPS aggregate and 30 % of POFA as a replacement of cement.

Overall, OPS concrete is an environmentally friendly product that has similar durability properties to the conventional lightweight concretes (Teo et al., 2009), with higher water absorption and lower workability and strength (Itam et al., 2016).

POFA, another potential waste by-product has proven to have high amounts of silica and could be incorporated as a binder. It was first used as a partial replacement of cement by Awal and Hussin (Awal & Hussin, 1997). Their research concluded that the POFA could be a promising binder, and the effectiveness of the binder will increase as the replacement level of cement by POFA rises. However, a negative trend was observed as the replacement level exceeded 40%. On the contrary, Muthusamy et al. (2019) reported that the mix with 10% POFA produced better mechanical strength compared to the higher percentages. The incorporation of the 10% POFA, enhanced the durability and lowered the water absorption of the product.

POFA was utilized in foamed concrete and found useful in reducing the cost of the products (Munir et al., 2015). The researchers replaced between 10 to 50% of the cement by POFA and concluded that despite a decline in the compressive strength trend, with 50% of POFA, the loss of strength was only 30 to 40%. Conversely, Lim et al. (2013b) reported an enhancement of the mechanical properties of foamed concrete with the utilization of 10 to 20% of POFA.

The effect of the presence of POFA in the geopolymerization process as a binder was studied by Salih et al. (2014) and showed a high reactivity along with a comparable density which resulted in a 28-day compressive strength of 32 MPa. The durability of POFA/FA/GGBS based geopolymer mortar greatly depends on the POFA content as with high volume of POFA, the freezing-thawing and wet-dry cycles performance of the mix was reduced while a good resistance to the sulfuric acid was observed (Huseien et al., 2018b).

Researchers have utilized another palm oil by-product, namely POC as lightweight fine and coarse aggregates (Kanadasan & Abdul Razak, 2015a). Further, by grinding

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POC, palm oil clinker powder (POCP) could be produced and used as partial cement replacement (Ahmmad et al., 2017).

2.4 Palm oil clinker

Palm oil clinker (POC) is derived from incinerating the palm oil shell and mesocarp fibres in palm oil factories. It is a lightweight material with an irregular, dark coloured, and porous structure. The mineralogical composition of POC mainly includes quartz and cristobalite as the main phases (Kanadasan et al., 2015). The chemical composition of POC collected from different mills is summarized in Table 2.1.

Table 2.1: Chemical composition of palm oil clinker powder derived from different sources

Chemical composition of palm oil clinker powder								
Reference	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO	MgO	P_2O_5	$SiO_2 + Al_2O_3 + Fe_2O_3$
(Nayaka et al., 2018)	60.3	5.8	4.7	7.24	3.3	4.2	3.8	70.8
(Karim et al., 2016)	62.8	3.4	6.5	10.5	6.9	3.5	0	72.7
(Ibrahim & Abdul Razak, 2016)	59.9	3.9	6.9	15.1	6.4	3.3	3.5	70.7
(Ismail et al., 2020)	55.4	2.2	10.8	17.7	7.1	2	4	68.4
(Hamada et al., 2019)	67.3	4.1	8.1	8.5	4	2.7	2.5	79.5
Average	61.14	3.88	7.4	11.808	5.54	3.14	2.76	72.42

As mentioned in ASTM C618-12a (ASTM International, 2012b), the sum of the chemical composition of Silicon dioxide, iron oxide, and aluminium oxide should exceed 70% for class F pozzolan. Table 2.1 shows that most of the references confirmed this criterion as well as the average shown in the table. This could affirm the potential of POCP to be utilized in concrete as a binder. Nayaka et al. (2018) investigated the replacement of cement with POCP up to 80% and reported that with 40% of replacement, 12.4MPa of compressive strength was achieved for cement-lime mortar. However, 30%

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replacement was the maximum level to reach the target of 30 MPa. Incorporating POCP as a binder in cement mortar reduces the mechanical properties of the samples. Karim et al. (2016) investigated the thermal activation of the POCP by putting it in the furnace with the temperature of between 300°c and 650°c for three hours. They reported that the compressive strength increased remarkably, which could be due to the removal of the fibres and organic carbon during the calcination process which resulted in a reduction of the porosities in POCP.

Besides the powder, POC has also proven to be a potential replacement for fine and coarse aggregates in terms of structural efficiency and resistance to chloride penetration (Abutaha et al., 2018; Kanadasan et al., 2015). Although researchers (Abutaha et al., 2016; Abutaha et al., 2018; Kanadasan & Abdul Razak, 2015a) have used POCS in the concrete derivatives, a proper methodology of mix design on utilizing POCS has not been proposed.

A study proposed a mix design for self-compacting concrete with the incorporation of POC (Kanadasan & Razak, 2014). The methodology behind the mix design was using particle packing method, which is explained further. The aggregates are put loosely in a container with a known volume. Then, room-temperature water is added to the container to fill all the voids inside the aggregates up to the top surface. The amount of water added to the container is counted as the void inside the aggregates and this method will be used to replace the aggregates.

The positive thing about this method is considering the inner voids of the POC since it contains a considerable amount of porosities in it. Nevertheless, since the aggregates are put in a loose condition, the way that the aggregates are put together could vary considerably depending on the edges of the aggregates. As a result, due to the irregularity of the particles, as mentioned earlier in this section, the amount of added water could

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fluctuate. Moreover, the weight of the particles soaked in the water is not considered in the calculations, which is an influential factor in the calculation.

Sulaiman et al. (2020) investigated the effect of incorporation of palm oil clinker as partial sand replacement (10 - 40%) in concrete. The replacement of the sand with POCS was performed by weight percentage and the fresh properties of the mixes showed that the increase in the POC level decreases the workability of concrete. The mechanical properties of the mixes using 40% of POC demonstrated a reduction of around 20% compared to the control mix without POC.

Sharmin et al. (2015) studied the feasibility of using POC fine aggregate in geopolymer concrete with up to 50% replacement of the sand. POC was reported to be a suitable replacement for aggregates (Ibrahim & Abdul Razak, 2016) and it was utilized in the pervious concrete which caused a reduction of strength and density while the coefficient of permeability was increased. The obtained compressive strength with 100% replacement with POC was 3.4 MPa which is 65% of reduction to that of the control sample. Conversely, in another study (Abutaha et al., 2018), high strength concrete was developed using 100% POC and reported a reduction of 40% in the compressive strength compared to the control sample.

The incorporation of POC as the replacement of fine aggregate in self-compacting mortar showed that the density and the compressive strength of the mortar reduce as the replacement level increases. Furthermore, the carbon emissions of the mix containing POC showed a 50% reduction while benefitting from a cost reduction of around 13% compared to those of control mix (Kanadasan et al., 2018).

Overall, it can be concluded that the engineering properties of the products using industrial by-products greatly depend on the methodology of the research, mix design,

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and the materials. It is also worth mentioning that reusing these waste materials not only could lead to alleviating the disposal issue, but also would contribute to the preservation of natural resources. Table 2.2 summarizes the findings of previous researchers on utilizing POC and their significance.

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Table 2.2: Previous studies on palm oil clinker

Ref.	Binder content (kg/m^3)	Cement replacement with POCP	Type of sand	Sand replacement level	Cement-based material type	Methodology	Findings
(Kanadasan & Abdul Razak, 2015b)	860	0-50%	Conventional sand	0%	self-compacting mortar	POCP was used as cement replacement	POCP is suitable to use as a filler or binder material
(Kanadasan et al., 2015)	916-1018	NA[1]	POCS	100%	self-compacting mortar	POC from different states of Malaysia was compared	The differences between them are negligible, and most of them have satisfactory properties.
(Kanadasan et al., 2018)	900	NA	POCS	0-100% with increments of 12.5	self-compacting mortar	POCS in SSD condition was used for castings	POCS could decrease the CO_2 emissions and cost by 50% and 13%, respectively
(Kanadasan & Razak, 2014)	643-804	35-48%	POCS	0-100% with increments of 25	self-compacting concrete	particle packing method to determine the voids inside POC	Almost 68% of the strength could be achieved using the full replacement of POC compared to the control sample
(Kanadasan & Abdul Razak, 2015a)	605-791	NA	POCS	0-100% with increments of 25	self-compacting concrete	particle packing method to determine the voids inside POC	good strength and reduction in density can be achieved to produce satisfactory structural efficiency

Note: [1]NA stands for “not applicable”.

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Table 2.2, continued

Ref.	Binder content (kg/m^3)	Cement replacement with POCP	Type of sand	Sand replacement level	Cement-based material type	Methodology	Findings
(Ahmmad et al., 2017)	420-504	0-17%	Conventional sand	0%	lightweight concrete	POCP was used as a filler to decrease the voids inside the POC aggregates	With the addition of 15% of POCP to the cement, the compressive strength of 57MPa was achieved
(Abutaha et al., 2016)	420	NA	POCS	0-100% with increments of 20	lightweight concrete	Department of Environment method (DOE) was adopted for the mixture to produce Grade 40 concrete	28-day compressive strengths of 33-49 MPa were recorded, about 30 % reduction from the control sample
(Abutaha et al., 2018)	520	NA	POCS	0-100% with increments of 20	High strength concrete	particle packing method to determine the voids inside POC	28-day compressive strengths of 55 MPa was achieved compared to that of 92 MPa of the control sample

Note: [1]NA stands for “not applicable”.

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2.5 Geopolymer

One of the alternatives for producing concrete without cement is the geopolymer concept (Davidovits, 2008) which was first developed by Davidovits. Geopolymers are inorganic materials formed by the reaction process between an aluminosilicate source and an alkaline activator solution (AAS). With the utilization of geopolymers, waste materials and by-products can replace Portland cement, which could reduce the disposal issue and concerns regarding the CO_2 that is generated during the production of cement. Geopolymer concrete benefits from comparable mechanical characteristics (Aldred & Day, 2012) and enhanced durability properties (Albitar et al., 2017; Valencia Saavedra & Mejía de Gutiérrez, 2017) compared to the cement concrete. Various studies used different binders as aluminosilicate sources in geopolymers; fly ash (FA) (Gunasekara et al., 2019), metakaolin (MK) (Pouhet & Cyr, 2016), ground granulated blast-furnace slag (GGBS) (Patel & Shah, 2018), rice husk ash (RHA) (Kaur et al., 2018a), palm oil fuel ash (POFA) (Liu et al., 2016).

The most used alkaline solutions are sodium-based and potassium-based. A comparison of potassium-based and sodium-based fly ash geopolymer paste (Hosan et al., 2016) showed that sodium activator could achieve higher compressive strengths at ambient and elevated temperatures below 400 °C compared to potassium activator. On the other hand, potassium activator had a better strength at 600°C. The most used and effective activator is reported to be the combination of sodium silicate and sodium hydroxide (Tho-in et al., 2012). The geopolymerization process relies on the method of curing. With the increase in temperature, the geopolymerization accelerates and with longer curing time, the properties of the products will be enhanced (Ng et al., 2018). The samples are usually cured in the oven with temperature varying from 60 to 110°c for 24 hours. Among all the binders, FA is the most used widely one because of its abundance and resemblance to cement in terms of final properties. Low calcium FA (class F) has

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approved to be a potential binder while high-class FA (Class C) geopolymer concrete can produce a 65 MPa compressive strength (Chindaprasirt et al., 2007).

Geopolymers are mostly suitable for precast concrete elements since they mostly need to be cured at temperatures higher than the ambient. Some attempts were performed in order to eliminate the need for oven curing such as solar curing (Dong et al., 2017) and ambient curing (Islam et al., 2015). However, the outcome has not been practical widespread.

2.5.1 Alkaline activators

The most common alkaline activators used in the geopolymerization process are a combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) or potassium hydroxide and potassium silicate (K_2O_3Si).

Statistics showed that the alkaline activator solution is blamed for the majority of the total emissions in the geopolymer concrete (Turner & Collins, 2013). However, the addition of slag could lower the need for the activator solution (Habert et al., 2011). Malkawi et al. (2016) investigated the effects of alkaline solution on properties of high calcium geopolymer mortars. They concluded that the NaOH concentration and Na_2SiO_3/NaOH ratio have a similar effect on the workability and setting time of the geopolymers mixes; increasing these ratios increases the setting times and reduces the workability.

Görhan and Kürklü (2014) studied the effect of the alkaline solution on the compressive strength of geopolymer mortar. They used Sodium hydroxide with 3, 6 and 9 molarity to cure the samples for 24 hours between 65-85 °C. Their result indicated that the highest compressive strength comes from curing under 85 °C using 6 M NaOH. One of the essential underlying facts in every research is decreasing costs. Alkaline activators

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are responsible for the majority of the cost. Some investigations focused on replacing sodium silicate by the low-cost waste glass (Duxson et al., 2007; Torres-Carrasco & Puertas, 2014).

In the production process of sodium silicate, quartz and sodium carbonate are heated to a high temperature of up to 1500℃ for the calcination, which produces large amounts of CO_2 in the air (Rodríguez et al., 2013). Sodium silicate is considered as an energy-intensive product that disputes the sustainability of using geopolymer concrete as an alternative to conventional concrete. Moreover, according to the current production of sodium silicate, the replacement of conventional concrete would not be feasible as the currently available sodium silicate may only provide less than 1% of the annual need of geopolymer concrete (Assi et al., 2020).

Another application of sodium silicate is in the batik industry. Batik is a traditional technique that was originated in Indonesia and is used for dyeing textiles on various materials. It is most well-known in Southeast of Asia and considered as one of the first cottage textile manufacturing in Malaysia that currently accommodates thousands of active factories (Birgani et al., 2016). Sodium silicate is used as a stabilizer of the dye colours to make them more durable. Nevertheless, a large amount of batik waste is produced from the washing process of the clothes, which contains sodium silicate. Not many studies have been conducted on the generated batik waste and its treatment (Birgani et al., 2016; Mukimin et al., 2017; Sutisna et al., 2017) due to the complexity of the procedure.

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CHAPTER 3: MATERIALS AND METHODOLOGY

3.1 Introduction

This chapter presents the proportion of materials used and the experimental testing procedure of the current study. A wide range of tests were performed to analyse and investigate the properties of the mortar and concrete.

3.2 Volume-based approach

In most studies, the design and replacements are performed using the weight-based approach. Considering the fact that not all the materials have the same physical properties, the weight-based approach does not consider the volume of the materials. The volume-based is adopted in this study based on specific gravity of the materials.

With the weight-based replacement that has been used widely, the volume of the POC is increased in the mixes due to its lower specific gravity. This results in high binder content in order to compensate for the increased volume of the sand. Since the sand acts as the filler in the mortar, with the change in the volume of the sand, the content of the binder should be relatively changed. Nevertheless, with the volume replacement, the amount of binder used in the mix is not affected much since the volume of the sand is kept constant.

3.3 Research process flow chart

The flowchart of overall research process and steps followed in the current research work is shown in Figure 3.1. The experimental tests performed in this study were chosen to investigate different dimensions of the effect of sand replacement in cement and geopolymer mortar. A great emphasis has been on the high absorption rate and porosity level of POCS in conducting different type of tests.

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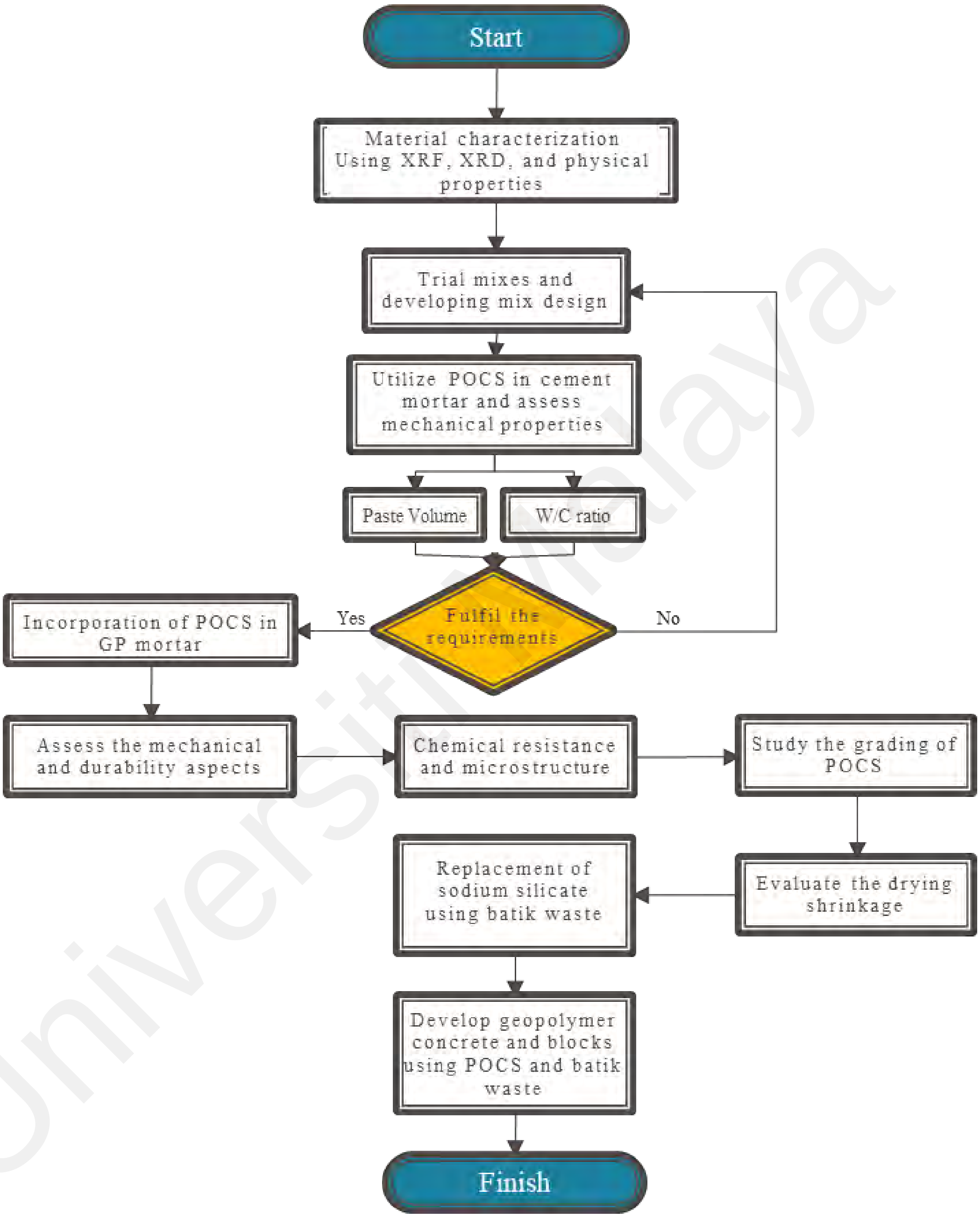

Figure 3.1: The research process flowchart

3.4 Materials

The main components of geopolymers are the binders and alkaline activators.

3.4.1 Binders

Ordinary Portland cement (Type I) with a strength class of 42.5 MPa and a specific gravity of 3.15 was used for the cement mortars and control mixes. For geopolymer mixes, class F fly ash was used with a specific gravity of 2.4, and it was used as the control binder in geopolymer concrete in this research. FA was replaced partially by GGBS with a specific gravity of 2.75, to tackle the need for oven curing of geopolymer samples. Incorporating GGBS could expedite the setting of the mix and increase the early strength development due to the presence of high amount of calcium.

Chemical composition of the materials was analysed using X-ray Fluorescence (XRF) and illustrated in Table 3.1. As can be seen, POC has the highest SiO_2 content which shows its potential to be used as sand. It is also noted that GGBS has 43.3% of calcium oxide.

Table 3.1: Chemical composition of materials

Chemical composition	OPC	POC	FA	GGBS
SiO_2	16.86	59.90	58.3	29.3
Al2O3	3.83	5.37	22.5	14.2
Fe_2O_3	3.76	6.93	8.0	0.5
CaO	68.14	6.37	5.2	43.3
MgO	2.04	3.13	0.9	7.8
P_2O_5	0.04	0.07	0.4	-
K_2O	0.21	15.1	1.8	0.48
SO_3	4.84	2.60	0.6	2.1
TiO_2	0.14	0.12	1.2	0.84
MnO	0.11	0.12	0.1	0.32
Na_2O	0.03	0.24	0.6	0.6
LOI	2.01	-	0.4	0.6

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3.4.2 Fine and coarse aggregates

Mining sand (MS) and crushed granite were used as the control fine and coarse aggregates (in the concrete specimens), respectively and replaced with POC. The XRF results show a high amount of silica (SiO_2) in the POC as the main constituent of sand is also silica, usually in the form of quartz. Based on the previous XRF test results on 12 POC samples collected from different states of Malaysia (Kanadasan et al., 2015), the silica content of POC ranged from 57.4 to 74.3%, confirming high silica content in POCS of the present research. POC was collected from Sime Darby Plantation factory in large pieces with 50 to 150 mm in size. It was then crushed and sieved in the laboratory to obtain the size of below 4.75 mm for sand and between 4.75 to 15 mm for coarse aggregate as natural fine and coarse aggregate replacements. POC has a porous nature, and it is a lightweight material.

POCS was obtained from Sime Darby Plantation factory with irregular shaped porous pieces of about 50 to 200 mm in size and processed in the laboratory with the size of below 4.75 mm to be used as fine aggregate and between 4.75 to 15 mm for coarse aggregate. They are acquired from the incineration of oil palm shell (OPS) and the fibre in specific proportion. POC has porosities both on the surface and inside.

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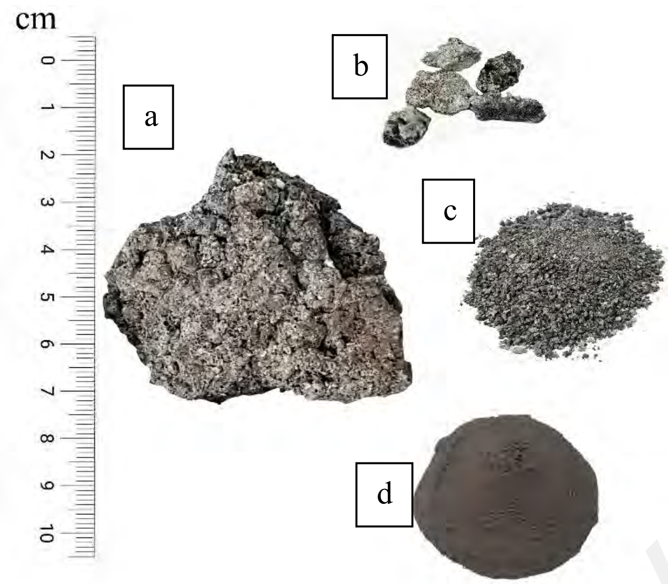

Figure 3.2: a) POC chunk collected from the factory, b) POC coarse aggregate, c) POC fine aggregate, d) POC powder

Throughout this research, POCS was compared to MS in different aspects and castings. Physical properties of POCS and MS is depicted in Table 3.2. As can be seen, the fineness modulus of both sands is almost similar while there is a substantial difference in water absorption and moisture content. Besides, the specific gravity of POCS is noted much lower to that of MS.

Table 3.2: Physical properties of MS and POCS

Physical Properties	MS	POCS
Particle size (mm)	≤ 4.75	≤ 4.75
Fineness modulus	3.43	3.52
Specific gravity	2.65	1.92
Water absorption (%)	0.7 ± 0.15	3.3 ± 1
Moisture content (%)	0.2 ± 0.06	1.5 ± 0.5
Bulk density (kg/m^3)	2.05	1.60

Sieve analyses were performed on MS and POCS following ASTM C136/C136M (ASTM International, 2014a) to calculate the fineness modulus of the sands by dividing

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the sum of the cumulative percentage retained on the sieves by one hundred. Figure 3.3 shows the particle size of MS and POCS, in which the particle size for both sands falls in the well-graded aggregate category according to the British Standard 882:1992 and the result shows similar trends in their particle sizes which could be beneficial for the comparison of the sands. The fineness modulus of POCS and MS was calculated as 3.52 and 3.43, respectively.

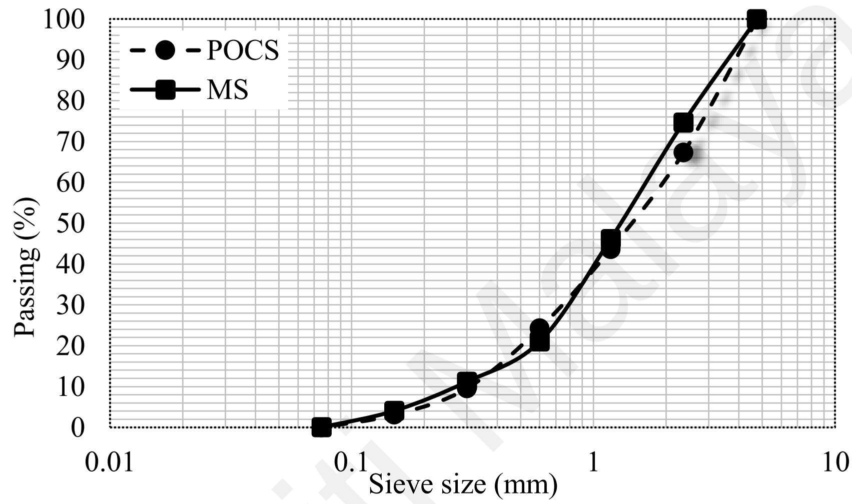

Figure 3.3: Sieve analysis on MS and POCS

In order to have a better understanding of the physical appearance, the Scanning Electron Microscope (SEM) images of both MS and POCS was examined with a magnification of 1000x and shown in Figure 3.4. MS shows pores in micro scale while POCS possesses pores with size ranging from 25-100 micrometres. Higher porosities of POCS in comparison with MS, increases the surface area and the water absorption of the sand particles. Further effects of the porous nature of POCS is discussed in Chapter 4.

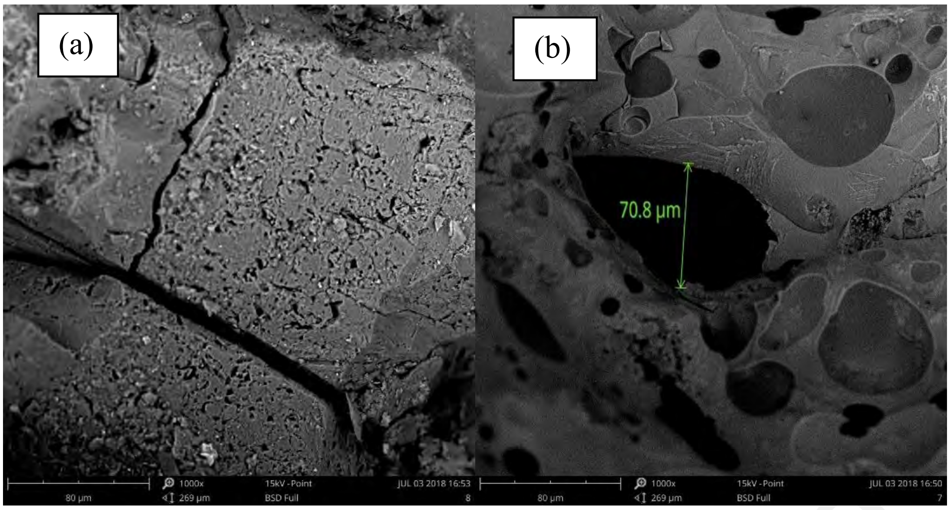

Figure 3.4: SEM images of a) MS, b) POCS

3.4.3 Alkaline activators

A combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) with SiO_2/Na_2O ratio of 2.44 was used in this study for the geopolymerization of the binders. The mass ratios of sodium silicate to sodium hydroxide and AAS to the binder of 1.5 and 0.5, respectively, were used in all the mixes.

3.4.4 Batik waste

Batik industry in Malaysia is one of the booming cottage industry that starts with the drawing on the white clothes and chanting process to create patterns with wax (Figure 3.5). Next, the clothes are coloured using Remazol or Naphthol dyes and in the current research, the waste from Remazol dye that was used as silicate source (Figure 3.6). To stabilize the colours on the clothes, they are soaked inside the sodium silicate solution using two methods of manual soaking and using an automated soaking machine (Figure 3.7).

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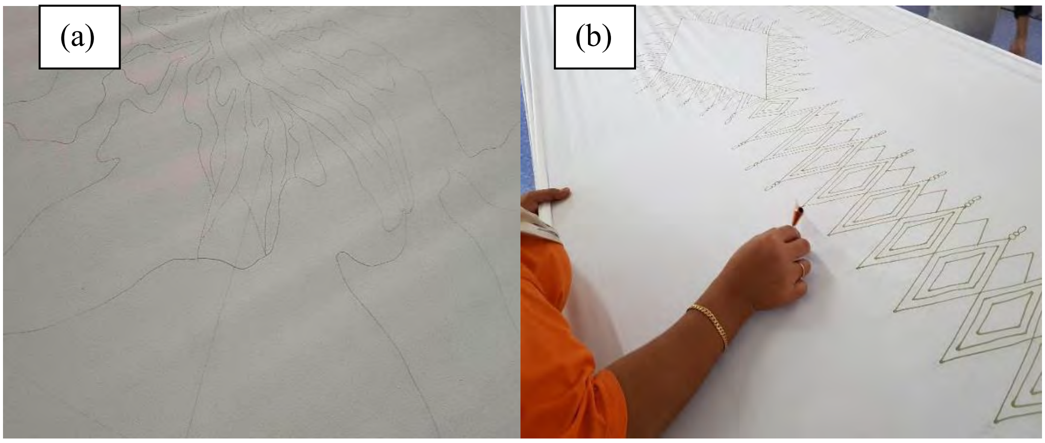

Figure 3.5: a) batik drawing on the white clothes, b) batik chanting using wax

Figure 3.6: Batik colouring of the clothes

Figure 3.7: Batik clothes soaking step using an automated soaking machine

The sodium silicate used in the batik factories is the commercial C140. Then, the clothes are left to dry for 3 to 4 hours prior to the washing stage with cold water. In order

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to remove the wax off the clothes, a mixture of sodium carbonate and hot water is usually used subsequent to the cold-water washing. The whole process is shown in Figure 3.8.

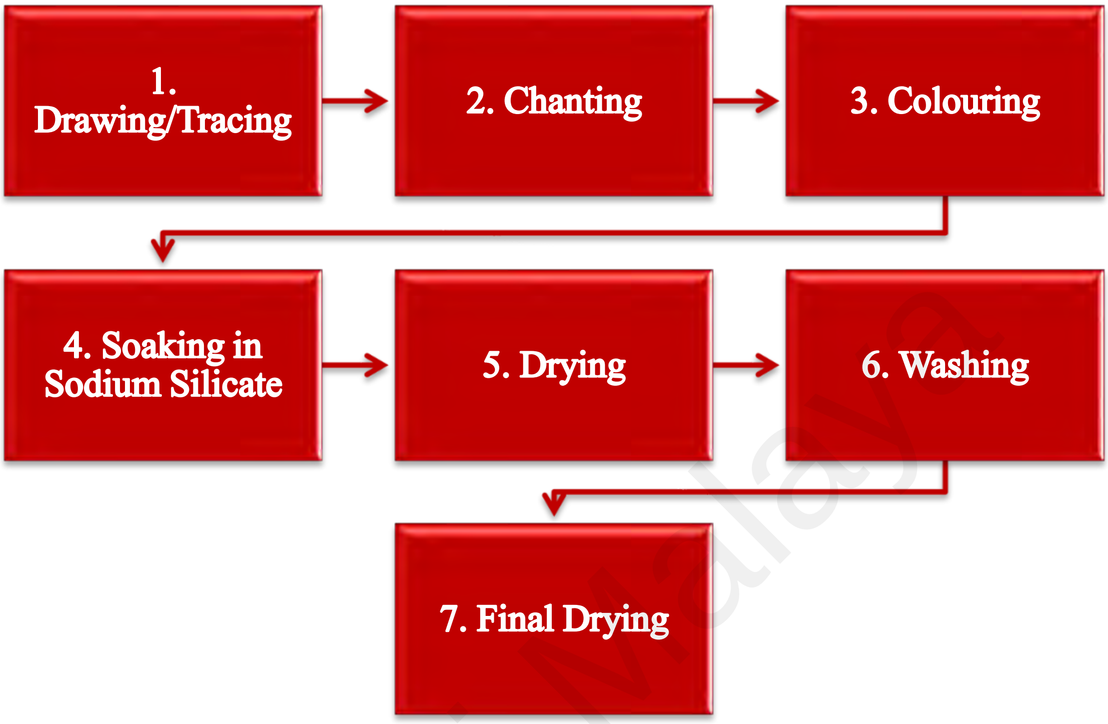

Figure 3.8: Batik process in Malaysia

The excess of the sodium silicate solution is washed off from the clothes during the washing process. This batik waste could have adverse effects on the environment due to the presence of chemicals. Thus, by utilizing the batik waste (Figure 3.9) in the geopolymer concrete as a silicate and water source, it could not only have an economic justification, but it could also benefit the environment by diminishing the amount of batik waste to be released in the environment.

Figure 3.9: Batik waste

The batik waste was analysed through the inductively coupled plasma optical emission spectroscopy (ICP-OES) test using the Perkin Elmer Optima 8300 equipment. This test method was performed to confirm the presence and quantity of sodium silicate by checking the sodium (Na) and silica (Si) elements in the sample. The samples were collected from the batik waste generated during the washing phase of the clothes from the factory and was diluted in deionized water to get different concentrations for verification purpose. The ICP results showed that the batik waste contains 5024 ppm of Si and 4826 ppm of Na. It is worth mentioning that small amounts of aluminium, magnesium, and calcium were also noticed in the result.

3.5 Mixing procedure

In order to ensure homogenous mixes, dry mixing procedure was performed with both bowl mixer and manual hand mixing. Dry mixing was performed for 4-5 minutes, followed by 4 minutes of wet mixing. As for geopolymers, Sodium hydroxide solutions were prepared one day before the mixing day and mixed with the sodium silicate 5 minutes before commencing the mixing. NaOH and sodium silicate need to be stirred and diluted thoroughly to gain a consistent result.

After mixing, the mortar and concrete were cast into 50 mm and 100 mm cube moulds, respectively, with two layers of compaction using the vibrating table. Specimens were

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demoulded after 24 hours and then placed for their respective curing condition until the day of testing; cement samples were put in the water tanks and geopolymers in the oven and ambient condition depending on their curing method.

3.6 The experimental tests

The codes of practice related to each test conducted throughout this research along with the sample requirements is summarised in Table 3.3.

Table 3.3: Summary of the codes of practice used throughout the study

Properties	Test	Reference	Sample Type	Number of Samples
Material properties	Specific gravity of binders	ASTM C188-15	-	-
	Specific gravity of fine aggregates	ASTM C128-15	-	-
	Sieve analysis of fine aggregates	ASTM C136-14	-	-
Fresh properties	Flow test	ASTM C1437-15	-	-
	Slump test	BS EN 12350-2	-	-
Mechanical properties	Compressive strength of mortar	ASTM C109-16a	50-mm cube	3
	Hardened density	ASTM C642-13	50-mm cube	3
	Setting time	ASTM C807-13	-	-
	Sorptivity	ASTM C1403-15	50-mm cube	3
	Water absorption	ASTM C642-13	50-mm cube	3
	Drying shrinkage	ASTM C1148	Prism	5
	Flexural strength of mortar	ASTM C348	Prism	3
	Acid resistance	ASTM C267	50-mm cube	3
	Sulfate resistance	ASTM C1012	50-mm cube	3
	Compressive strength of concrete	BS EN 12390-3	100-mm cube	3
	Splitting tension test	BS EN 12390-6	Cylinders	3
	Flexural strength of concrete	BS EN 12390-5	Prism	3
	Ultrasonic pulse velocity (UPV)	ASTM C597	50-mm cube	3
Microstructure	X-Ray Fluorescence	-	-	-
	X-Ray Diffraction	-	-	-
	Scanning electron microscope (SEM)	-	-	-

3.6.1 Flow test

The flow test was performed on the fresh mortar in accordance with ASTM C1437-15 (ASTM International, 2015c). The fresh mortar was poured into the flow table mould and compacted in two layers using the flow test tamper. The flow was measured in four defined lines after 25 drops of the table, and the average diameter was taken.

3.6.2 Compressive strength

The compressive strength test was carried out in accordance with ASTM C109-16a (ASTM International, 2016a) at the age of 1, 7, 28, 90, and 180 days. The cement-based cubes were in a saturated surface dry condition at the time of testing, while the geopolymer-based specimens were in the ambient condition. The compressive strength was calculated using the failure load divided by area of the specimen. Three specimens were tested with the average value being reported.

3.6.3 Density

The saturated surface dry (SSD) density of the cube specimens was calculated using the weight and dimensions of the cubes immediately following their removal from the curing tank after wiping off the surface moisture. The average of three cubes was taken for each age of testing.

3.6.4 Water absorption

The water absorption rate of the samples with a w/c ratio of 0.50 was investigated, and the average of three cubes for each mix at the age of 28 days was reported in accordance with ASTM C642-13 (ASTM International, 2013a). The specimens were dried at a temperature of 110°C for at least 24 hours to remove the moisture, and the weight of the cube specimens was measured. The cubes were subsequently immersed in the water for at least 24 hours, and the percentage of the amount of moisture absorbed by the specimens was calculated.

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3.6.5 Rate of water absorption (Sorptivity)

The test was conducted in accordance with the guidelines of ASTM C1403 - 15 (ASTM International, 2015b)

3.6.6 Setting time

The setting time test procedure was carried out in accordance with ASTM standard test method C807-13 (ASTM International, 2013b) using the modified Vicat needle.

3.6.7 Drying shrinkage

For evaluation of the drying shrinkage of the mixes, five prism specimens of 25x25x285 mm for each mix were prepared and cured according to the ASTM C1148 (ASTM International, 2014d) specifications, and the length change of the specimens was monitored for 30 weeks. A digital strain gauge reader with an accuracy of 0.1 μm was used to monitor the length change of the mortar prisms between two demec points installed on two sides of the prisms. The average of the two sides for each specimen was taken prior to taking the average of the five specimens for each mix.

3.6.8 Flexural strength of mortar

In order to evaluate the flexural strength of the mixes, three prism specimens of 40x40x160 mm were cast and tested at the age of 28 days. The procedure for the test was in accordance with the ASTM C348 (ASTM International, 2014b) in which a three-point loading with a load rate of 0.5 mm/min was applied.

3.6.9 Splitting tensile of concrete

The splitting tensile test was performed in accordance with BS EN 12390-6. Three cylinders were tested using a jig at the age of 28 days and the average was reported.

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3.6.10 Acid resistance

The procedure of the test was performed in line with ASTM C267-01 (ASTM International, 2012a) guidelines. Hydrochloric acid was used with a concentration of 3% to evaluate the performance of the mixes against chemical attack. The acid solution was changed every 28 days throughout the testing period of 180 days to ensure the effectivity of the solvent.

Prior to the immersion of the cubes in the acid, the weight, dimensions, and physical appearance of the cubes were recorded to monitor the changes that would occur due to chemical attack. The compressive strength of the cube specimens was determined at 28-, 56-, 90-, and 180-day after immersion. The physical appearance and weight loss, however, was monitored every seven days. The specimens were cleaned through rinsing in running water three times, followed by a quick dry between each rinse. Afterwards, the cubes were left to dry for 2 hours before the physical inspection and strength evaluation were carried out.

3.6.11 Sulfate resistance

ASTM C1012 (ASTM International, 2015a) was used as the reference to test the specimens with a 5% concentration of magnesium sulfate ($MgSO_4$) with a pH value between 6 and 8. The solution was changed every 28 days, and the compressive strength of the cubes was measured at the ages of 28, 56, 90, and 180 days after immersion in the sulfate solution.

3.6.12 Ultrasonic pulse velocity (UPV)

The UPV test was conducted in accordance with ASTM C597 (ASTM International, 2016b) using 50 mm cube specimens. Prior to the test, the machine was calibrated each time using the reference cylinder provided with the apparatus. In order to ensure the stability and accuracy of the results, a grease-based coupling agent was applied to the

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specimens and transducers. The velocity was calculated by dividing the distance between the two transducers by the transit time. The average of three cube specimens was taken and reported.

3.6.13 X-ray Powder Diffraction (XRD)

The samples were powdered after 28 days of curing and then dried at a temperature of 110°C for 24 hours prior to the test. PANalytical XRD equipment with a Cu-Kα 1.54 Å radiation (45 kV, 30 mA) was used to analyse the samples at the 2θ range from 5 to 90 degrees at a step size of 0.026° and 150 s/step.

3.6.14 Scanning Electron Microscope (SEM)

The SEM images were taken using high-performance Phenom ProX desktop SEM equipment with an accelerating voltage of 15 kV and a resolution of less than 8 nm with high depth field. In order to examine the sufficiency of the cement paste in the mortars, the specimens were collected from the 28-day compression test samples to analyse the interfacial transition zone (ITZ) on the bond of both the MS and POCS mortars.

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CHAPTER 4: PROPERTIES OF CEMENT AND GEOPOLYMER MORTARS USING POCS

4.1 Developing POCS cement mortar

This study was conducted to compare the POCS with MS in the standard mortar. POCS occupies a larger volume with the same weight compared to the MS due to its smaller specific gravity. POCS is a very porous material which makes it lightweight and water-absorbent. In the previous studies conducted on POCS in cement mortar (Kanadasan et al., 2015; Kanadasan et al., 2018), high amount of binder was utilised to compensate for the porous particles of POCS using weight-based replacement approach, while the current study practices a volume-based approach to reduce the binder content. The proportions for standard mortar, as stated in ASTM C109/C109M (ASTM International, 2016a), were used as the reference mass proportions with 1: 2.75: 0.485 for cement: sand: water, respectively. These values were then converted to volume, based on their respective specific gravities to replace the sand with the same volume of POCS. The volume proportions occupied by the cement, sand, and water would be 1: 3.27: 1.53 (17.2%, 56.4%, 26.4%), respectively.

Since the water absorption of POCS is higher than the conventional aggregates (Abutaha et al., 2016), water/cement (w/c) ratios of 0.50, 0.55, and 0.60 were used for the POCS mixes to compensate for the difference. An initial w/c of 0.55 was used for the calculation:

$$\frac{Mass_{(water)}}{Mass_{(cement)}} = \frac{Density_{(water)} \times Volume_{(water)}}{Density_{(cement)} \times Volume_{(cement)}} = 0.55$$

The paste volume was kept constant. Thus, the sum of the cement and water by volume would be:

$$Volume_{paste} = volume_{cement} + volume_{water} = 17.2 + 26.4 = 43.61\%$$

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With the above-mentioned equations, the volume of cement and water would result in 15.96% and 27.65%, respectively, which results in the volume proportions of 1: 3.53: 1.73. Thus, the cement: sand: water mass ratio after the conversion to mass for the POCS mix would be as follows: 1: 2.16: 0.55. For the mixes with w/c ratios of 0.50 and 0.60, the same cement and sand ratios were implemented with the water content being the variation.

The mass proportions of the materials for the standard mortar and related POCS are given in Figure 4.1.

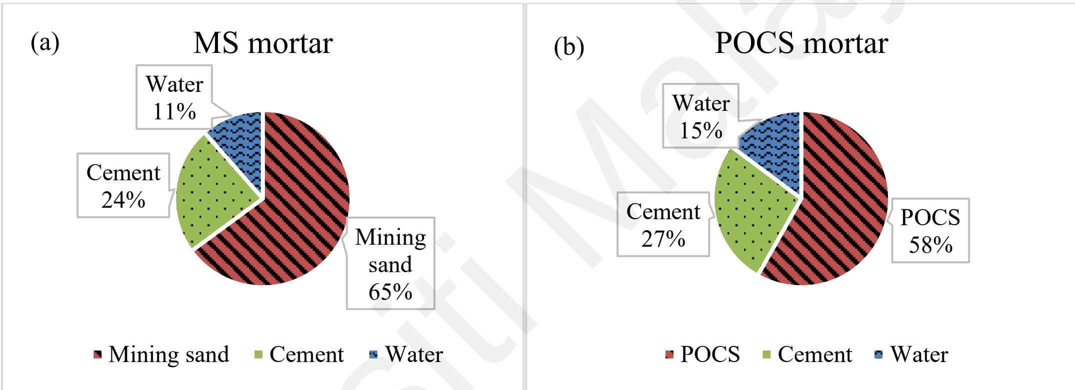

Figure 4.1: Mass percentages of the same volume of POCS and MS mortars

It is clear that with the same volume of POCS and MS, there is a noticeable decrease in the mass percentage of POCS due to its lower specific gravity. The mass percentages of POSC and cement paste were found to be 58.2 and 41.8, respectively.

To verify the performance of this mixing procedure, different POCS-cement paste percentages were tested and analysed. With the right amount of cement paste and w/c ratio, the porosities in the POCS can be filled with the paste which could lead to enhancement of the interfacial transition zone. Cement paste volume has a significant role in the properties of the concrete and mortar. Not only it has effect on the workability but also on the characteristics of the final product.

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In this experiment, three w/c ratios: 0.50, 0.55 and 0.60 were implemented for each cement paste percentage to ensure efficiency and to observe its effect on filling the POCS voids. It is worth mentioning that according to the ASTM, w/c ratio of regular mortar is 0.485, which was used for the control sample. For ease and accuracy of castings, after replacing POCS in the calculations, the volume percentages were back calculated to mass percentages. Table 4.1 shows the mix Proportions of the 25 mixes that have been cast.

Table 4.1: Mix proportions of POCS cement mortar

Mix ID	Cement paste (%)	Sand (%)	w/c ratio	Mix proportion (kg/m3)			
				MS	POCS	Cement	Water
M0	35	65	0.485	1494.4	-	543.4	263.6
M1	39	61	0.50	-	1151.8	490.9	245.5
M2	41	59	0.50	-	1113.0	515.6	257.8
M3	42	58	0.50	-	1093.6	528.0	264.0
M4	43	57	0.50	-	1074.3	540.3	270.1
M5	45	55	0.50	-	1035.6	564.9	282.4
M6	47	53	0.50	-	997.0	589.4	294.7
M7	49	51	0.50	-	958.4	613.9	307.0
M8	51	49	0.50	-	920.0	638.4	319.2
M9	39	61	0.55	-	1139.5	470.0	258.5
M10	41	59	0.55	-	1100.5	493.4	271.4
M11	42	58	0.55	-	1081.1	505.1	277.8
M12	43	57	0.55	-	1061.6	516.7	284.2
M13	45	55	0.55	-	1022.9	539.9	297.0
M14	47	53	0.55	-	984.2	563.1	309.7
M15	49	51	0.55	-	945.7	586.2	322.4
M16	51	49	0.55	-	907.2	609.2	335.1
M17	39	61	0.60	-	1128.2	450.8	270.5
M18	41	59	0.60	-	1089.1	473.0	283.8
M19	42	58	0.60	-	1069.5	484.1	290.4
M20	43	57	0.60	-	1050.1	495.1	297.1
M21	45	55	0.60	-	1011.2	517.1	310.3
M22	47	53	0.60	-	972.5	539.0	323.4
M23	49	51	0.60	-	934.0	560.9	336.5
M24	51	49	0.60	-	895.6	582.6	349.6

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4.1.1 Flow test of cement mortar

Figure 4.2 shows the results of the flow tests in which the control MS mortar with a flow of 65% is shown as a single point in the chart. As observed, for w/c ratios lower than 0.60, low flow measurements were recorded for the mixes with sand percentages of 59 and 61%. The workability increased with the increase in the paste volume (Chu, 2019) and w/c ratio (Li et al., 2019a). Paste volume is considered as a vital factor in fresh and hardened concrete (Piasta & Zarzycki, 2017) as it directly correlates with the coating thickness of the aggregate particles. The maximum flow was found to be well above 110%.

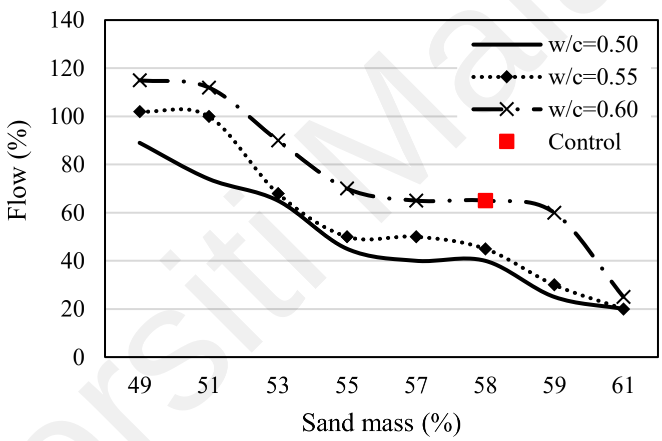

Figure 4.2: Flow test results of three w/c ratios of POCS cement mortar

The flow of the control mixture was recorded as 65% with a w/c ratio of 0.485, while the flows for the same volume for the POCS were recorded as 40, 45, and 65% for w/c ratios of 0.50, 0.55, and 0.60, respectively. It can be derived that the workability of the MS mortar with a w/c ratio of 0.485 is equivalent to the workability of POCS mortar with a w/c ratio of 0.60. Ramappa Ramesh et al. (2018) used a similar w/c ratio (0.58) for POCP in masonry mortar to get the desired flow. The need for water content increases with the increase in the pore structure of the mortar (Venkatarama Reddy & Gupta, 2008). As shown in Figure 3.4, POCS has a lot of pores inside and on the surface, which could lead to water absorption from the water that was meant for hydration. Moreover, the physical property test on water absorption showed that POCS absorbed about 3.3%. The relation between the paste content, w/c ratio, and flow percentage is shown in Figure 4.3.

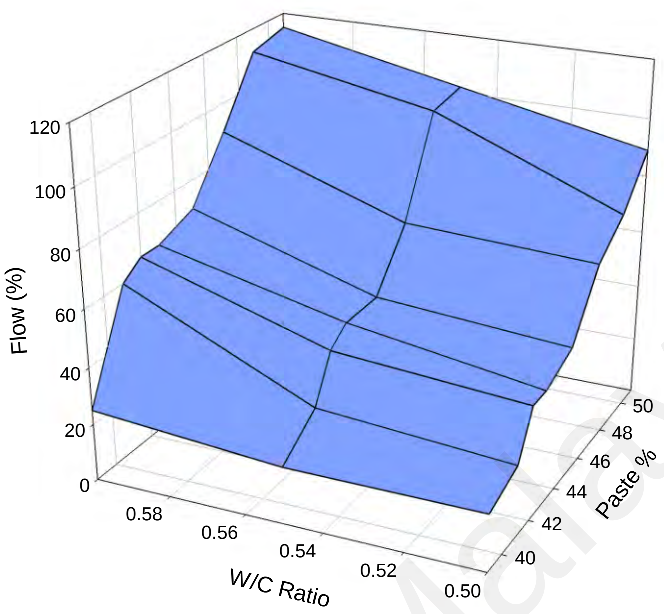

Figure 4.3: Relationship between paste content, w/c ratio and percentage of flow of POCS cement mortar

4.1.2 Density of cement mortar

The saturated surface dry (SSD) densities of the cube specimens are shown in Figure 4.4. The porous nature of the POCS makes it lightweight, with a concomitant decrease in the density of the mortar (Karim et al., 2017). In the mixes with a sand content higher than 58%, the specimens were not compacted efficiently, even though the vibrating table was used to compact the mixes in two layers for all the mixes. As observed by visual inspection, this was due to insufficient cement paste to fill the voids between the sand particles. A research conducted by Li et al. (2019a) on the effect of a low w/c ratio on the porosity content and strength development has shown that an increase in the w/c ratio causes the porosity to decrease and the bulk density to increase.

The 28-day density of the POCS mortar specimens ranged between 1845 and 1913 kg/m^3 compared to 2173 kg/m^3 for the control specimen. The lower the sand percentage, the higher the density becomes, albeit with a gradual increase in the slope. The advantage of utilising POCS instead of MS is evident in the reduction in the SSD, which was recorded between 12 and 15% for the mortars prepared with a sand mass of 49-58%; however, a reduction of up to 29% was obtained in the density for the sand mass that was higher than 58%, albeit with lower strength due to the low paste content. This trend is in line with the findings from other researchers (Kanadasan et al., 2015; Kanadasan et al., 2018) who reported a 7-11% reduction in the density with the utilisation of POCS. The smaller reduction compared to the results from the current research could be attributed to the relatively higher binder content.

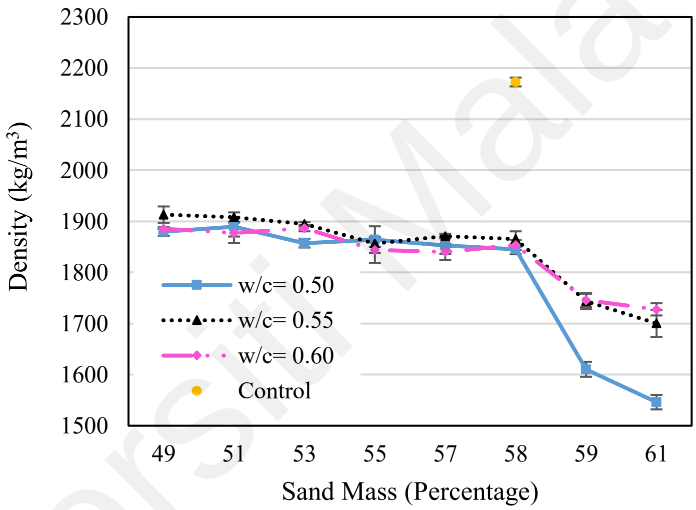

Figure 4.4: 28-day SSD density of POCS cement mortar

4.1.3 Compressive strength of cement mortar

The compressive strength of different w/c ratios at the ages of 1, 7, and 28 days are shown in Figure 4.5, Figure 4.6, and Figure 4.7. The strength of the control mix is shown as a reference point in the graphs. The 28-day compressive strength of the control mix was recorded at 47.1 MPa. In contrast, the specimen with 100% replacement of MS with POCS for the same volume, achieved a lower compressive strength of 41.18 MPa; a reduction of about 12.5% compared to the control mix. This can be reduced to 9.7% with an increase of 5% in the cement paste for POCS mortar. The maximum strength was achieved at 53% of sand mass for w/c ratios of 0.50 and 0.55, and 55% of sand mass for a w/c ratio of 0.60. A comparison with the literature (Kanadasan & Abdul Razak, 2015a; Kanadasan & Razak, 2014) shows that a similar trend in the reduction in the compressive strength was observed as the 28-day strength was reduced between 20 and 30% with the full replacement of sand with palm oil clinker even though a high amount of binder content was utilised (600 – 800 kg/m^3).

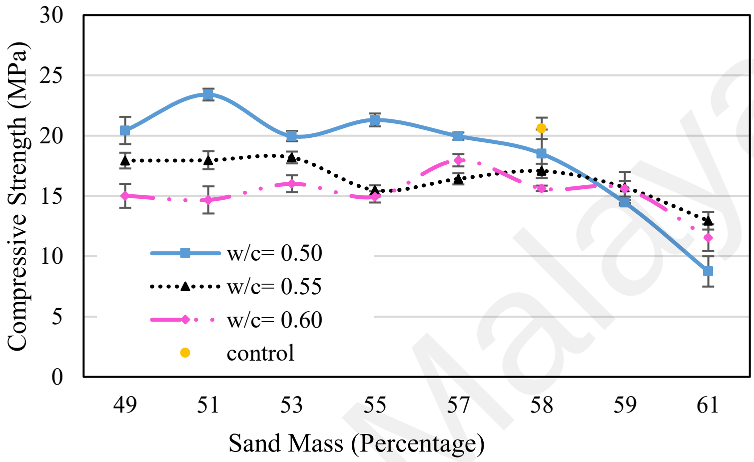

Figure 4.5: 1-day compressive strength of POCS cement mortars

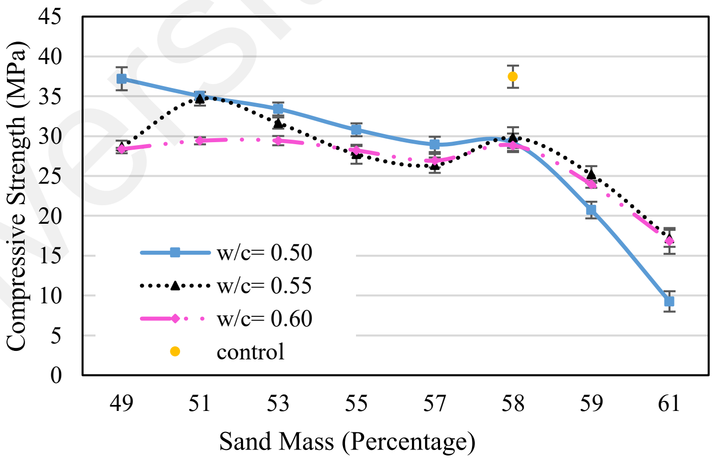

Figure 4.6: 7-day compressive strength of POCS cement mortars

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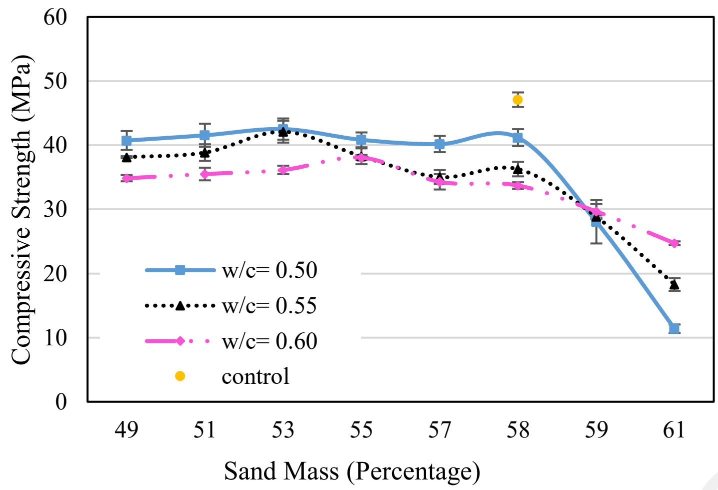

Figure 4.7: 28-day compressive strength of POCS cement mortars

For quantities of sand mass higher than 58%, mixes with higher w/c ratios of 0.55 and 0.60 achieved higher compressive strengths, which could be attributed to the dryness of the mixes coupled with insufficient paste in the lower quantities of paste mass, while for the sand mass percentages between 49 and 57, higher strength was achieved for a w/c of 0.50.

The mixes with 53 to 58% of sand mass with a w/c ratio of 0.50 produced comparable compressive strengths. However, lower sand content resulted in higher workability. Reducing the sand mass to less than 53% (51% and 49%) had a negative effect on the strength of the mixes for all the w/c ratios. There was a steady decrease in the strength as the sand mass decreased indicating that the paste content is relatively high in proportion to the sand layers, and, hence, increasing the cement paste beyond this point would result in capillary pores as the fine particles increase. Moreover, shrinkage cracks would appear as a result of the heat of hydration of the cement paste leading to lower strength (Piasta & Zarzycki, 2017; Rozière et al., 2007). With the increase in the paste content, the thickness of the cement paste-fine aggregate interface increases which could diminish the adhesion of the paste to the sand particles (Piasta & Zarzycki, 2017), and, ultimately, decrease the strength of the specimens as the bond weakens. The compressive strengths and hardened SSD density of the samples are given in Table 4.2.

Table 4.2: Compressive strengths with respective densities cement mortar

Mix ID	Compressive strength (MPa)			Density (kg/m^3)
	1-day	7-day	28-day	
M0	20.61	37.45	47.09	2172.8
M1	8.74	9.26	11.39	1546.1
M2	14.44	20.72	28.05	1610.4
M3	18.50	29.17	41.18	1845.6
M4	19.96	28.96	40.17	1852.7
M5	21.31	30.80	40.83	1876.0
M6	19.96	33.40	42.51	1891.4
M7	23.41	35.04	41.54	1909.5
M8	20.43	37.20	40.73	1913.1
M9	12.95	17.18	18.32	1700.2
M10	15.71	25.19	28.85	1744.0
M11	17.07	29.81	36.27	1865.1
M12	16.42	26.35	35.03	1871.3
M13	15.48	27.73	38.27	1872.7
M14	18.19	31.64	42.10	1894.9
M15	17.96	34.69	38.85	1908.2
M16	17.93	28.64	38.13	1913.2
M17	11.54	16.85	24.71	1727.7
M18	15.60	23.96	29.67	1745.4
M19	15.63	28.84	33.72	1853.4
M20	17.96	26.92	34.29	1840.0
M21	14.92	28.20	38.07	1844.0
M22	16.01	29.44	36.14	1884.5
M23	14.67	29.41	35.51	1882.47
M24	15.02	28.38	34.85	1887.48

Figure 4.8 shows the failure mode of cube specimens under compression, which was investigated using a microscope. Bond failure occurs when the fine aggregates are detached from the cement paste, while the splitting of the sand particles is caused by sand failure. After examining several samples from the M3 mix at the age of 28 days, it could

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be seen that sand failure and bond failure between the POCS, and the cement paste occurred at the same time. This could confirm that POCS has the required stiffness and strength to be used as sand.

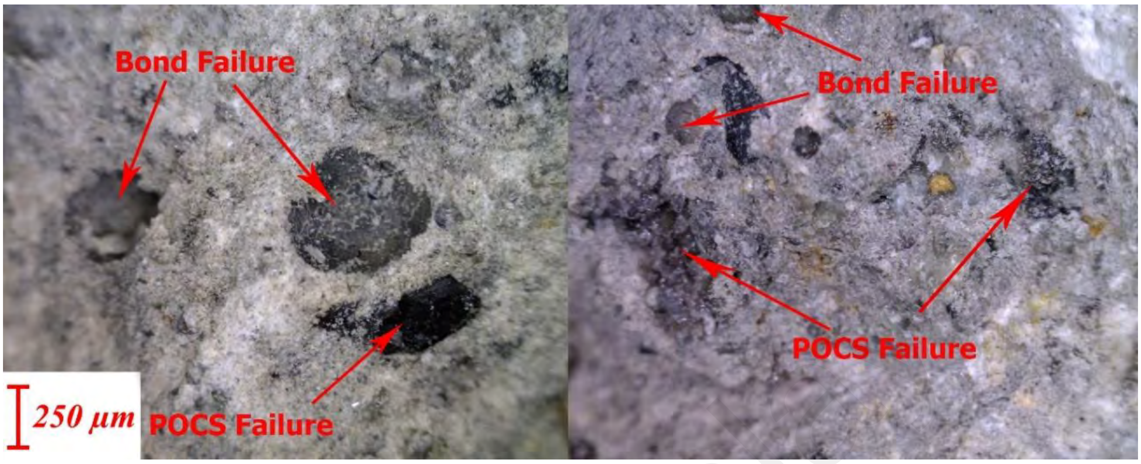

Figure 4.8: Microscopic view of failure modes of cement mortar specimens

4.1.4 Compressive strength Development of cement mortar

Figure 4.9 shows the percentage of the compressive strength developed in the first 24 hours divided by the 28-day compressive strength. Overall, similar strength development was observed for POCS specimens to that of the control MS mix (shown as a single point). Nevertheless, the mixes with a w/c of 0.50 gained higher strength development of 45 to 77% while the control mix achieved 44%. This higher early strength development could be attributed to the higher porosity of the POCS as it increases the surface area and results in higher pozzolanic activity (Wei Chen, 2007). Moreover, the larger surface area and higher internal curing of POCS, could lead to the prevention of microcracks (Ahmmad et al., 2017).

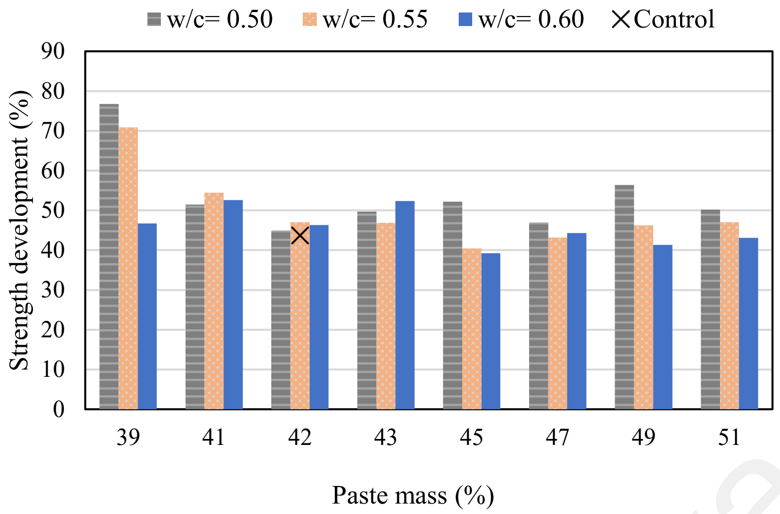

Figure 4.9: Strength development of 1-day/28-day with w/c of 0.50

4.1.5 Effect of w/c ratio on the Strength

In the cement-based mortar, the effect of the w/c ratio is vital in the strength development of the specimens. As a result, the w/c ratio for the relative sand percentages is reviewed based on the data in Figure 4.7.

The consistency and uniformity of all the specimens improved with the increase in water content; the higher strengths were achieved for specimens with a w/c ratio of 0.50 compared to those of 0.55 and 0.60 for sand contents between 49 and 58%. A similar trend was observed by another study (Haach et al., 2011), which was performed to evaluate the effect of the w/c ratio on the properties of mortar. Increasing the water content between 59 and 61% of sand mass increased the strength, as the mixes with lower paste masses were fairly dry, and, due to the high sand volume, the aggregates were not coated sufficiently with the paste. Thus, with higher w/c ratios, the excess water was used for the lubrication of aggregates and filling of the voids, which contributed to the strength development; a similar increase in the compressive strength was reported with an increase in the w/c ratio (Li et al., 2019a). Figure 4.10 shows the relationships between the paste content, w/c ratio, and the 28-day compressive strength of all the mixes.

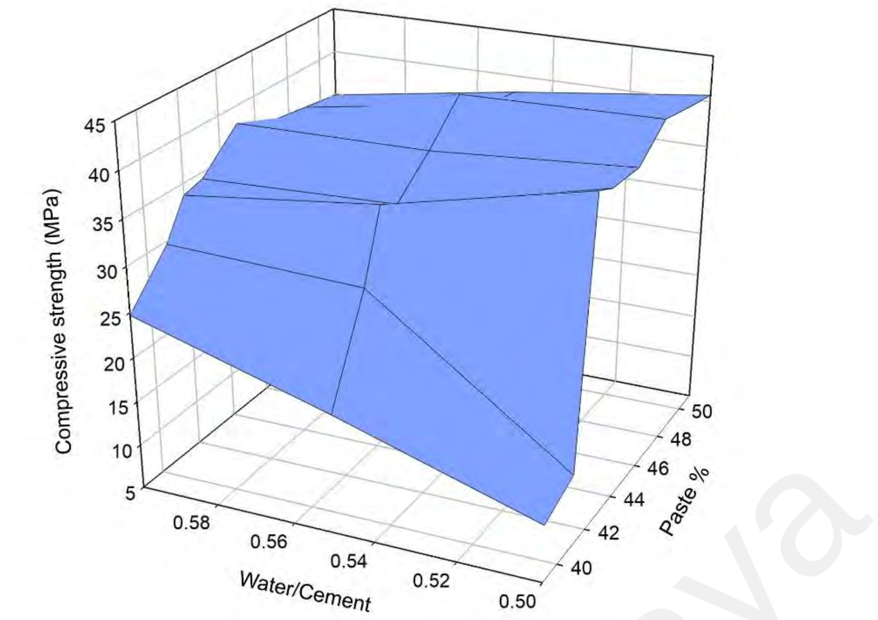

Figure 4.10: The relationship among paste content, w/c ratio, and 28-day compressive strength

4.1.6 Ultrasonic pulse velocity of cement mortar

The UPV result can demonstrate the quality, uniformity, cavities and the compactness of cement paste and sand. Figure 4.11 represents the effect of sand content on the UPV for different w/c ratios. UPV values of 3.66 to 4.58 km/s could represent good quality (Hwang et al., 2012). The 28-day UPV result of all the tested samples surpassed the minimum range for good quality specimens. At 53% of the mass percentage and a w/c ratio of 0.50, a UPV of 4.09 km/s was recorded, which placed it in the good quality range. The velocity of the control sample was found to be 4.41 km/s at 28 days, which is slightly higher than the POCS mortar, and is shown in the figure as a single point. Previous studies (Abutaha et al., 2016; Kanadasan & Razak, 2014; Kanadasan et al., 2018) confirmed the reduction in the UPV values with the incorporation of POCS in the mixes due to the voids in the POCS and the impeding effect of air.

For all the w/c ratios, the trend of the UPV results was similar; the highest UPV values belonged to the 53-58% of sand content. The lower values in the 61% and 59% of sand mass could be ascribed to the unfilled pores since the paste content was not adequate for

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the mixes. It is reported that shrinkage has a direct relation with the paste volume; increasing the paste could cause cracks to appear (Piasta & Zarzycki, 2017) due to the increased degree of cement hydration. This could justify the decreased UPV values for the sand content of less than 53%.

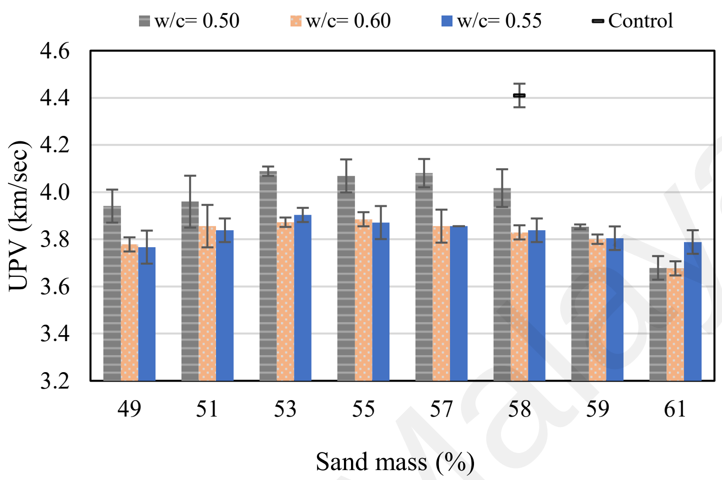

Figure 4.11: 28-day ultrasonic pulse velocity of cement mortar specimens

Figure 4.12 depicts the relationship between UPV and the 28-day compressive strength of the mortars. Previous studies (Hamid et al., 2010; Kewalramani & Gupta, 2006) have shown that UPV correlates well with the compressive strength. The related strengths and UPV values of the POCS mixes show that the specimens with higher UPV value will demonstrate higher strength. The correlation coefficient of the equation is found to be 0.91, which indicates the reliability of the equation. As a result, this equation can be employed to predict the strength of the POCS mortar specimens based on their respected UPV value, and, since the UPV is a non-destructive test, the compressive strength could be predicted without the need for crushing the specimen.

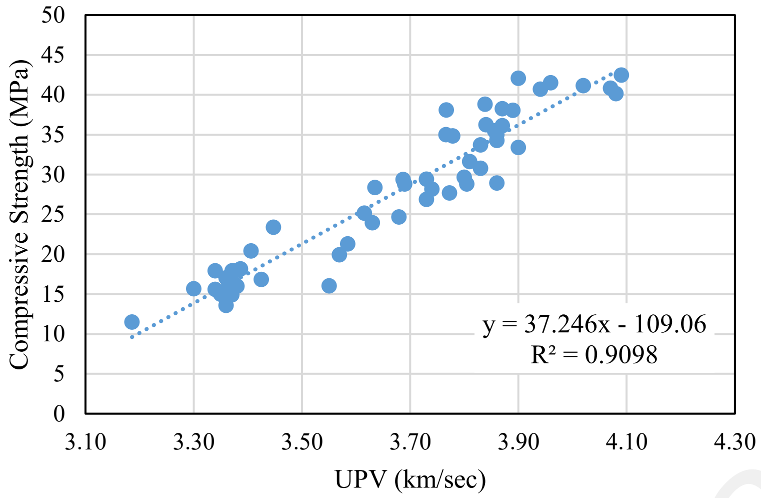

Figure 4.12: Compressive strength and ultrasonic pulse velocity correlation of POCS cement mortar

4.1.7 Water absorption of cement mortar

Water absorption performance has a crucial impact on the durability; it is mainly related to the porosity level of the specimens, which could be enhanced by the increased pozzolanic activity (Aslam et al., 2017; Pachideh & Gholhaki, 2019). The values for the water absorption of the specimens are given in Figure 4.13. Increasing the cement paste caused a reduction in water absorption due to the filling of the POCS porosities. This could be supported by the increase in the density, as shown in Figure 4.4. Cement paste tends to fill the pores, which will subsequently lead to a denser structure, and, once the pores have been filled, the bond between the cement paste and the sand particles in the ITZ will be enhanced, thereby resulting in a lower water absorption value. The water absorption of the control mix is shown in the graph as a single point recorded as 5%, while the POCS mixes experienced a water absorption of 6.8 to 9.5%. According to a study (Neville, 2018), water absorption rates of less than 10% could be considered as high-quality concrete, and, according to the results, the mixes containing a sand content of less than 58% are eligible for this category. The resistance of the specimens toward water penetration could be ascribed to the secondary CSH gel that creates a denser composition (Ahmad Zawawi et al., 2020).

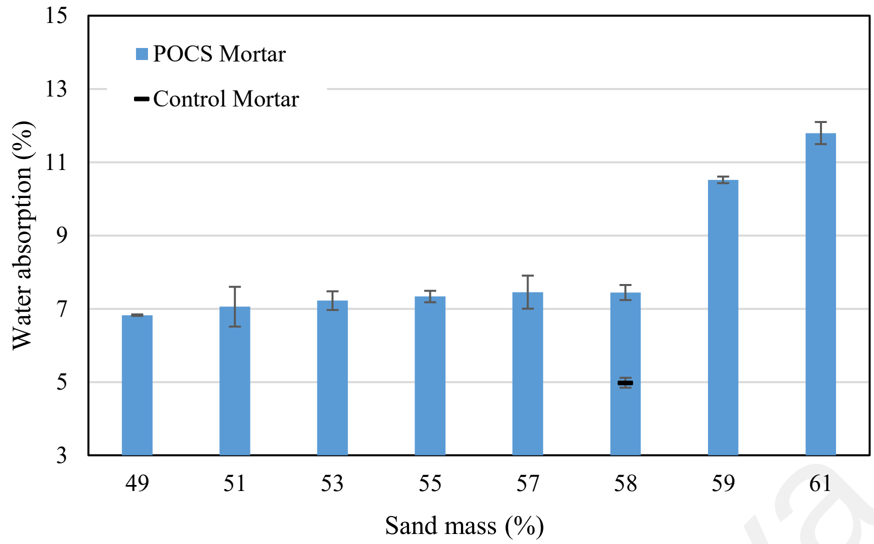

Figure 4.13: Water absorption of POCS specimens in comparison with the control mixture

4.1.8 XRD analysis of cement mortar

The microstructural effect of the w/c ratio on the cement paste was studied using XRD, and, for this purpose, diffraction patterns for cement paste using three w/c ratios were prepared and analysed after 28 days of curing, as shown in Figure 4.14. Mortar mix M3 was studied in addition to the cement pastes through XRD peaks. The intensities of the pastes slightly reduced with the increase in the w/c ratio, which reveals that the excess water can be effective in degrading the reactivity of the cement paste. This could be an outcome of a lower hydration peak. Previous studies showed no change in the heat generated by the Portland cement hydration with a change in the w/c ratio (Kirby & Biernacki, 2012; Pang & Meyer, 2016), while the evidence of the reduced peaks can be argued as being due to the reduced space for the reaction process, and the dendritic nature of the hydration products (Bishnoi & Scrivener, 2009; Scherer et al., 2012). The evidence of the Portlandite ($Ca(OH)_2$) was found at 18.1, 34.1, 50.8, 54.3, and 62.4 degrees, which is a vital mineral for C-S-H gel and a dominant factor in respect of the properties of cement paste.

According to the peak pattern of the M3 mortar mix, Quartz was a match at 22 and 26.7 degrees with high intensities confirming high silica content of the mortar (Figure 4.14). The reduction in the Portlandite level after adding POCS could be due to the reactivity of the POCS. The Quartz present in the POC was reported to be reactive with the amorphous phase (Karim et al., 2017). Although the composition of different sands varies, silica (SiO_2) is the most common component of sand, mainly in the form of Quartz, which is responsible for its considerable hardness.

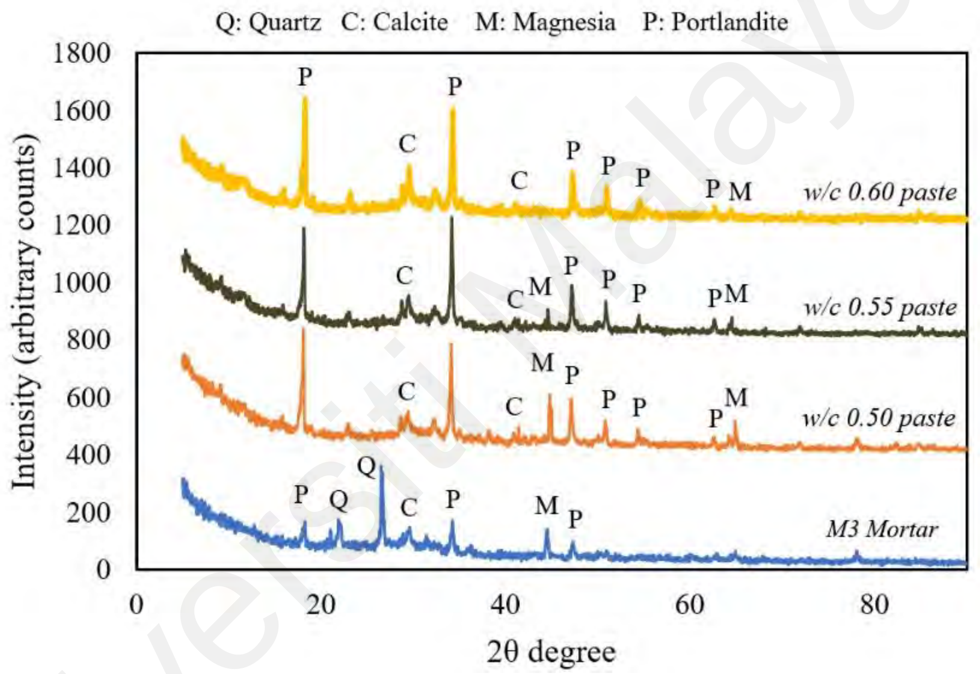

Figure 4.14: XRD on cement pastes with different w/c ratios and M3 mortar

4.1.9 Scanning Electron Microscope (SEM) of cement mortar

Scanning Electron Microscope (SEM) images were used to investigate the ITZ on the bond of both the MS mortar and POCS mortar. As observed in the mechanical properties, the mixes with paste contents of less than 42% produced lower strength compared to the other mixes. Thus, three mixes were chosen to evaluate the bond and the interaction between the sand and cement paste; M0 as the control sample with MS, M1 with 39% paste as representative of low strength, and M3 with 42% paste as representative of a high strength mix. The images are depicted in Figure 4.15 with a magnification of 1000x.

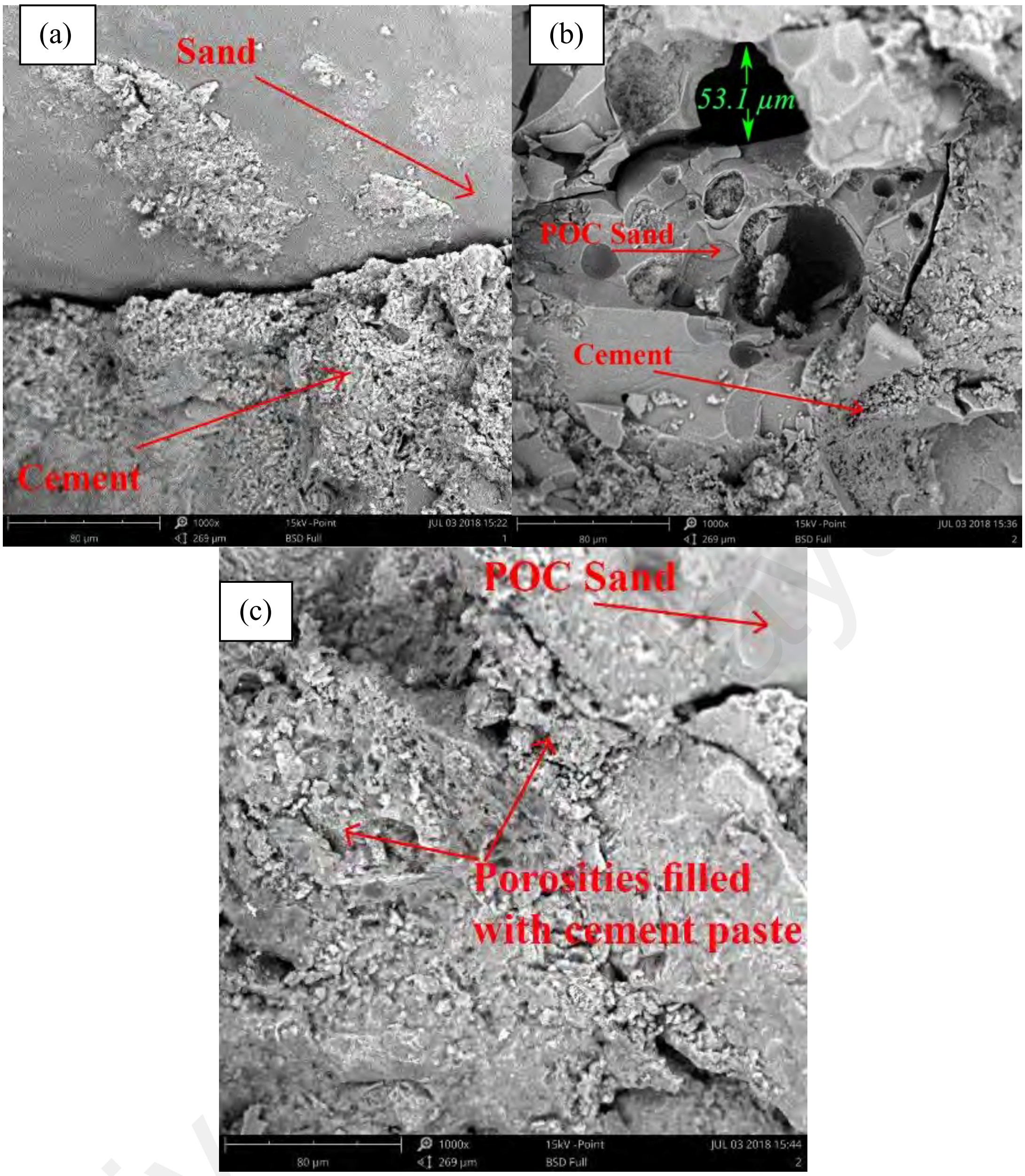

Figure 4.15: SEM images of cement mortar specimens: a) M0, b) M1, c) M3

Figure 4.15 (a) shows the bond between the MS and cement paste in the control sample (M0). MS has a smooth surface covered with cement paste. Figure 4.15 (b) unveils the unfilled layers of 41% (M1) of POCS; the inadequacy of the paste in comparison with the sand is clear. On the other hand, Figure 4.15 (c) displays the mix with 42% (M3) of cement paste, in which the cement paste is sufficient in combination with POCS. Generally, the voids in the POCS were filled, thereby improving the bond, and, as a result, the overall performance of the cement paste.

4.1.10 Sustainability of cement mortar

The increase in industrial wastes is compounding the issue of disposal and environmental pollution, and, hence, the significance of sustainability through the utilisation of such potential industrial by-products and industrial wastes is vital. As known, both the environment and the economy are the main aspects of sustainability. Moving towards sustainability has been one of the main concerns of the construction sector in terms of environmentally friendly resources and the replacement for natural resources (Kanadasan & Abdul Razak, 2015a). The abundance of palm oil mill by-products and wastes, especially in Southeast Asia, and its versatility would enable the construction industry to infuse sustainable materials through the use of POCS in making lightweight concrete.

Damineli et al. (2010) have introduced an index of binder intensities (bi) to measure the efficiency of the required binder for producing 1 MPa of strength. For this purpose, the binder content of the mix (kg/m^3) is divided by the acquired strength at 28 days. The (bi) index has been calculated along with the total cost of 1m^3 of all the mortar mixes in Malaysia and shown in Table 4.3. Furthermore, the cost of each mix divided by the 28-day compressive strength is shown in Table 4.3 as a cost index, which demonstrates the cost needed for producing 1 MPa of strength.

The control mix produced the lowest (bi) index among all the mixes, while M3 produced the lowest (bi) and cost indices using POCS. This shows that the mix with 42% of paste mass and 58% of POCS has shown to be the most sustainable mix in terms of low binder usage and economic view among the POCS mixes. Based on the estimation, the costs of 1 m^3 of M0 and M3 mixes are 47.7 and 40 USD, respectively. This suggests that with the incorporation of POCS in the mortar, about a 16% reduction in the price of mortar is expected.

The (bi) index of the M0 and M3 mixes are calculated as being 11.5 and 12.8 kg m^{-3} MPa^{-1}, respectively. The comparison between these values shows that with 100% replacement of the conventional sand with palm oil clinker sand, the binder content experiences a slight increase in the binder (10%) while incorporating large amounts of waste materials that use the minimum required energy could result in sustainable construction (Luis de Oliveira Júnior et al., 2019; Naik, 2008). The (bi) indices of the POCS mortar in the previous studies was calculated as 13 (Kanadasan et al., 2015) and 13.4 kg m^{-3} MPa^{-1} (Kanadasan et al., 2018), which are slightly higher than M3 mix. Thus, it could be envisaged POCS as a potential and sustainable alternative material.

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Table 4.3: (bi) index and cost comparison of MS and POCS cement mortars

Mix ID	Cost (USD/m^3)	Cost/strength (USD/MPa)	bi index (kg m^{-3} MPa^{-1})
M0	47.7	1.01	11.5
M1	38.2	3.36	43.1
M2	39.4	1.41	18.4
M3	40.0	0.97	12.8
M4	40.6	1.01	13.5
M5	41.8	1.02	13.8
M6	43.0	1.01	13.9
M7	44.2	1.06	14.8
M8	45.4	1.11	15.7
M9	36.9	2.01	25.7
M10	38.0	1.32	17.1
M11	38.5	1.06	13.9
M12	39.1	1.12	14.8
M13	40.2	1.05	14.1
M14	41.3	0.98	13.4
M15	42.4	1.09	15.1
M16	43.5	1.14	16.0
M17	35.6	1.44	18.2
M18	36.7	1.24	15.9
M19	37.2	1.10	14.4
M20	37.7	1.10	14.4
M21	38.8	1.02	13.6
M22	39.8	1.10	14.9
M23	40.8	1.15	15.8
M24	41.9	1.20	16.7

Note: the price of the materials in Malaysia (USD/kg): Cement 0.06, MS 0.01, palm oil clinker sand 0.0075, water 0.0005

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4.2 Utilization of POCS in geopolymer mortar

The behaviour of palm oil clinker sand (POCS) geopolymer mortar in terms of mechanical properties, durability, and microstructural was compared to that of with MS. Moreover, with the help of GGBS, the geopolymer mortar's dependency on the oven curing method was tackled. Flow test, compressive strength, ultrasonic pulse velocity, water absorption, sorptivity, setting time, chemical resistance tests along with the x-ray diffraction and energy dispersive x-ray analyses were performed to evaluate the performance of the mixes.

Different molar ratios of NaOH from 8 to 16 were prepared to investigate the effect of the NaOH's concentration on the properties of the POCS geopolymer mortar since the properties of the products immensely depend on the concentration of the activator's concentration. On the other hand, to study the effect of incorporation of GGBS, 10 Molar NaOH was used for the FA/GGBS mixes for economic justification.

A volume-based design was carried out for the replacement of the MS with POCS due to the porous nature of POCS. For this purpose, the specific gravity of the sand was used to calculate the volume of the MS and replace it with the same volume of POCS. A total number of 16 mixes were cast for the current study. In all the mixes, MS was replaced by POCS, and a FA-based geopolymer mortar with MS was used as the control design to compare the behaviour of the POCS with the MS in the geopolymer mortar. Different percentages of FA-GGBS were used with GGBS ranging from 10 to 50%. To achieve the flow of 110 ± 5 mm for all the mixes as recommended by the ASTM C109/C109M-16a (ASTM International, 2016a), additional water was added to the mixes to get the desired flow. The additional water can provide better consistency and longer setting time, especially for the mixes incorporating GGBS. Two different types of curing were used: oven curing at 65°c for 24 hours and ambient curing. The mix proportions are shown in Table 4.4.

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Table 4.4: Mix proportions of the POCS geopolymer mortar

ID	Binder (%)	Sand	Mol.	Curing	Sand (kg/m^3)	FA (kg/m^3)	GGBS (kg/m^3)	NaOH (kg/m^3)	Silicate (kg/m^3)
GP-1	FA (100)	MS	10	65°c for 24h	1367.2	645.3	0.0	129.1	193.6
GP-2	FA (100)	POCS	8	65°c for 24h	991.0	645.0	0.0	129.0	193.5
GP-3	FA (100)	POCS	10	65°c for 24h	991.0	645.0	0.0	129.0	193.5
GP-4	FA (100)	POCS	12	65°c for 24h	991.0	645.0	0.0	129.0	193.5
GP-5	FA (100)	POCS	14	65°c for 24h	991.0	645.0	0.0	129.0	193.5
GP-6	FA (100)	POCS	16	65°c for 24h	991.0	645.0	0.0	129.0	193.5
GP-7	FA (90) GGBS (10)	POCS	10	65°c for 24h	991.0	584.6	65.0	129.9	194.9
GP-8	FA (80) GGBS (20)	POCS	10	65°c for 24h	991.0	523.4	130.9	130.9	196.3
GP-9	FA (70) GGBS (30)	POCS	10	65°c for 24h	991.0	461.3	197.7	131.8	197.7
GP-10	FA (60) GGBS (40)	POCS	10	65°c for 24h	991.0	398.3	265.5	132.8	199.1
GP-11	FA (50) GGBS (50)	POCS	10	65°c for 24h	991.0	334.3	334.3	133.7	200.6
GP-12	FA (90) GGBS (10)	POCS	10	Ambient	991.0	584.6	65.0	129.9	194.9
GP-13	FA (80) GGBS (20)	POCS	10	Ambient	991.0	523.4	130.9	130.9	196.3
GP-14	FA (70) GGBS (30)	POCS	10	Ambient	991.0	461.3	197.7	131.8	197.7
GP-15	FA (60) GGBS (40)	POCS	10	Ambient	991.0	398.3	265.5	132.8	199.1
GP-16	FA (50) GGBS (50)	POCS	10	Ambient	991.0	334.3	334.3	133.7	200.6

4.2.1 Flow test of geopolymer mortar

One significant factor in defining the quality of the mortar and concrete is through checking the workability. The mixture should have sufficient workability for the transfer, placement, smooth surface finish (Kwasny et al., 2015) and performance (Alonso et al., 2017) with the minimum expenditure. All the mixes were designed to ensure a flow of

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110% ± 5 is obtained, and additional water of 5-17% of the binder's weight was added for all mixes depending on the requirement. Trial and error mixings were performed in order to get the sufficient flow of between 105% and 115%.

MS required less amount of water to produce the same amount of flow as POCS since POCS possesses a higher water absorption rate. Due to the higher surface area of GGBS (Islam et al., 2014), the mixes required more water compared to mixes with FA. Further, with the increase in the concentration of the molarity of AAS, more water was needed to achieve a flow of 110%. The workability of the mortar decreases with the increase of NaOH molarity (Huseien et al., 2018a).

Although all the mixes produced 110% of flow, as found through visual observation, not all the mixes had the same viscosity. Also, it is well known that due to high viscous AAS, the viscosity of fresh geopolymer mixes was found higher compared to the cement mortar for similar paste volume and flow. Moreover, the higher the GGBS amount, the more viscous due to geopolymerization and creation of calcium aluminate silicate hydrate (C-A-S-H) gel (Ye & Radlińska, 2017). The additional water could, in fact, increase the workability of the mixes if used in concrete. It is reported that the viscosity of the geopolymer decreases with the increase in water content (Koutník et al., 2020). The results of added water and flow are shown in Table 4.5.

Table 4.5: Flow test result and additional free water of geopolymer mortar

Binder (%)	Sand	Mol.	Free water (%)	Flow (%)
FA (100)	MS	10	5	110
FA (100)	POCS	8	12	114
FA (100)	POCS	10	12	112
FA (100)	POCS	12	12	112
FA (100)	POCS	14	14	110
FA (100)	POCS	16	14	105
FA (90) GGBS (10)	POCS	10	13	110
FA (80) GGBS (20)	POCS	10	15	110
FA (70) GGBS (30)	POCS	10	15	106
FA (60) GGBS (40)	POCS	10	17	113
FA (50) GGBS (50)	POCS	10	17	110

4.2.2 Effect of molarity of geopolymer mortar

Mixes GP-2 to GP-6, as shown in Table 4.4, were designed in such a way that the only variable among them was the molarity of NaOH. These mixes contained FA as the only binder with POCS as a 100% replacement for conventional MS as fine aggregate and cured in the oven. The comparison between the POCS and MS of the same molarity of 10 was also made in addition to the comparison among the mixes with different molarities.

The molarity of the NaOH correlates to the properties of the geopolymer mortar and increasing the molarity would results in improved dissolution to a certain amount depending on the precursor content and AAS ratio. Thus, study of the molarity of NaOH is an important step in the development of geopolymer.

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4.2.2.1 Compressive strength (molarity)

While demoulding the geopolymer cubes, it was found that the cubes were adhering to the moulds, which could be attributed to the cohesive nature of the sodium silicate and sodium hydroxide solution (Ma et al., 2018).

The control MS geopolymer mortar produced a 28-day strength of 50.5 MPa with a 10-molar NaOH solution. However, with the replacement of the MS by POCS, the 28-day strength varied from 19.8 to 41.3, with the NaOH molarity ranging from 8 to 16 (Figure 4.16). As expected, the strength of the specimens increased with an increase in the molarity of NaOH from 8-14 molar. However, increasing the molarity up to 16, had a negative effect on the strength. The comparison between the compressive strengths of the mixes with 14 and 16 molars shows a reduction of about 9 MPa for the latter. Researchers (Huseien et al., 2018a; Kaur et al., 2018a) reported that an increase in the molarity increases the mechanical properties of the specimens with the highest compressive strength achieved for mixes with 12 and 14 molars. When the concentration of the AAS becomes higher, the polycondensation is blocked by the acceleration in the reaction of the silica-alumina substance (Zuhua et al., 2009).

Moreover, it was reported by Lee and van Deventer (2002) that the reduction in the strength could be attributed to the precipitation of aluminosilicate gel caused by the redundant hydroxide ions. The optimum concentration of the NaOH depends on the binder type, alkaline activator, and curing conditions. An increase in the concentration beyond certain molarity is not recommended as it may have unfavourable effects (Wang et al., 1994) on the properties of the concrete, not to mention its economic aspect. With the same molarity of 10 (GP-1 and GP-3), the mix with POCS produced a compressive strength of almost 30 MPa while MS mixture produced 50 MPa. This reduction could be ascribed to the porous nature of the POCS with lower specific gravity compared to MS (Abutaha et al., 2016).

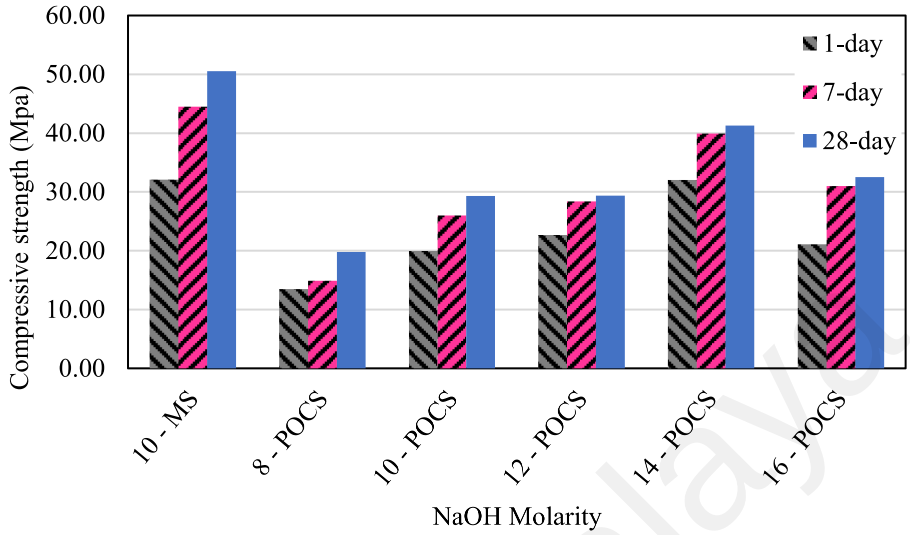

Figure 4.16: The effect of molarity on the strength of POCS geopolymer mortar

4.2.2.2 Ultrasonic pulse velocity (molarity)

UPV test is one of the means to check the quality of concrete in terms of uniformity, homogeneity, and determining the existence of internal voids and cracks According to the researchers (Hwang et al., 2012), a velocity of 3.66 to 4.58 km/s is classified as a good range of UPV value. A good UPV value denies the existence of large voids and porosities, and by maintaining the UPV in the satisfactory range, the structural integrity could be upheld (Kwan et al., 2012). Figure 4.17 shows the UPV results of the MS mortar along with the POCS mortar with different concentrations of NaOH. The 28-day test result of UPV of the control sample was found as 3.63, while the range for the POCS mixes was between 2.50 and 2.97 km/s. This decrease in comparison with the mix with MS is attributed to the porous POCS (Kanadasan et al., 2018). The POCS particles possess internal voids as well as external. With the penetration of the geopolymer paste, the outer voids of the sand particles would be filled. However, the trapped inner voids that were not filled with the paste could be the reason for the lower UPV values in POCS mortar specimens. Meanwhile, according to the trend of the UPV value in relation to the molarity of the NaOH, the mixes with higher molarities produced higher values of UPV. Higher concentration of NaOH increases the geopolymerization reaction, which could enhance the integrity of the specimens.

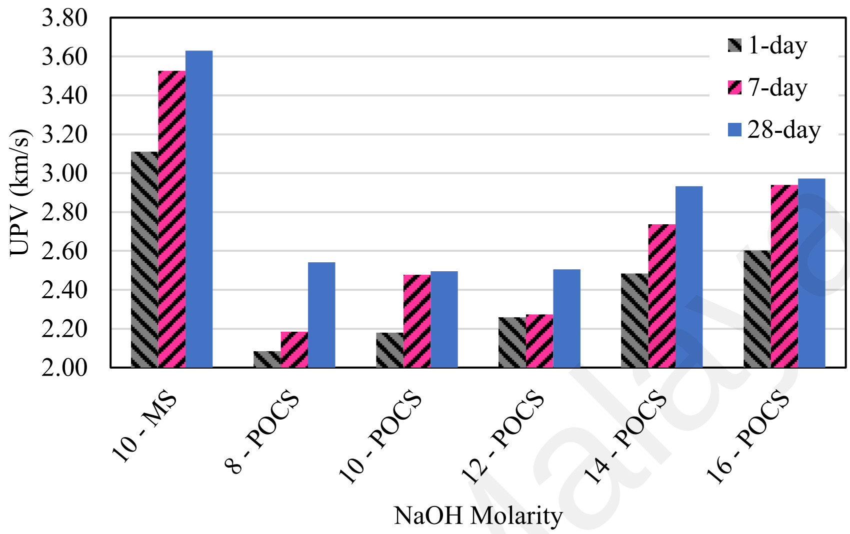

Figure 4.17: Ultrasonic pulse velocity for different concentrations of NaOH

4.2.2.3 Water absorption (molarity)

Based on the result of water absorption, the porosity of the cubes can be observed. Porosity is one of the essential properties which affects the durability of concrete. The water absorption test demonstrates the weight difference between the oven-dry and SSD condition of the specimens. In this study, the water absorption test was performed on the specimens at the age of 28 days. The results showed a decrease in the water absorption of the specimens with an increase in the molarity of the NaOH from 8 to 14-molar (Figure 4.18). An increase in the molarity level contributes to the geopolymerization process and improves the dissolution of binders because of the increased proportion of the sodium oxide to aluminium oxide and silica. The improved behaviour of the geopolymers with an increase in the NaOH molarity was also observed by other researchers (Bashar et al., 2014; Huseien et al., 2018a). Conversely, increasing the molarity to 16, increased water absorption. This could be ascribed to the decreased dissolution of the calcium, which brought about lower hydration products (Huseien et al., 2018a).

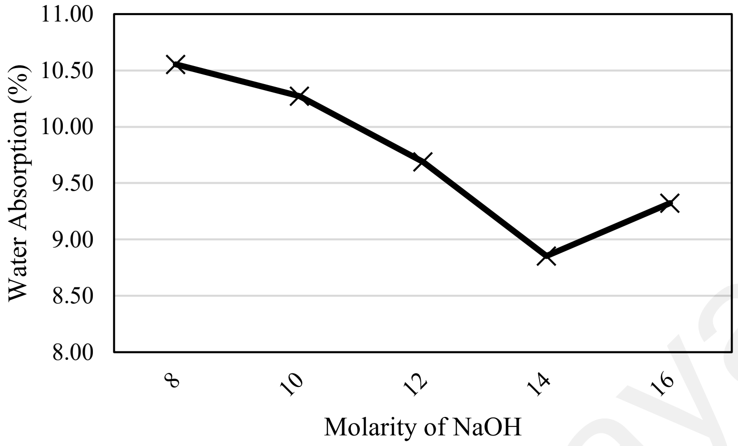

Figure 4.18: Effect of molarity on the water absorption

4.2.2.4 Rate of water absorption (molarity)

The water absorption rate of specimens in relation to time could reflect the capillary sorptivity, and it is an essential factor in the assessment of the durability. Figure 4.19 shows the rate of water absorption of geopolymer mortars with different molarities of NaOH. The results showed the same trend as the water absorption test; the highest rate belonged to the 8-molar mix while it decreased with the increase in the molar concentration. For the mix of 16-molar, however, a slight increase was found compared to the mix of 14-molar. The increase in the absorption rate of the mix with 16-molar NaOH could be attributed to the excess heat generated during the reaction that may have caused evaporation of a portion of the water. Thus, the absorption rate was observed slightly higher to that of the 14-molar mix. It was reported by Shaikh (2014) that sorptivity decreases with a higher concentration of NaOH, and it was concluded that with higher molar concentration and sodium silicate content, a considerable decrease in the sorptivity level was experienced and ascribed to the denser structure of the aluminosilicate gel.

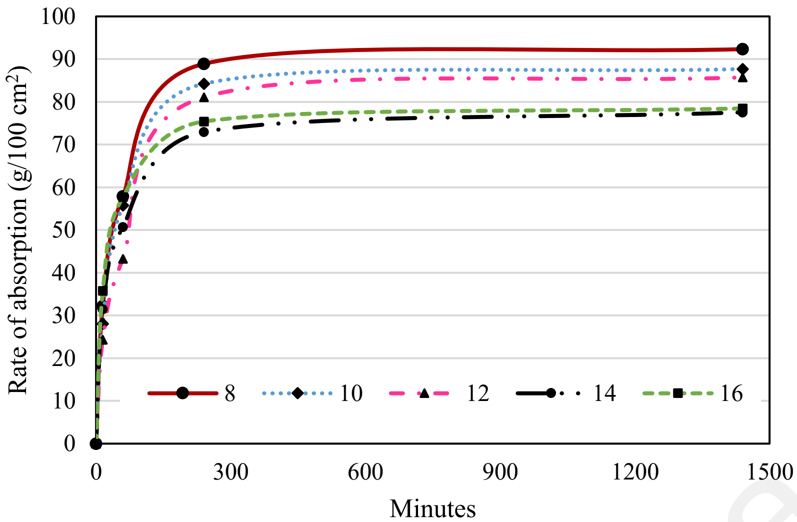

Figure 4.19: Effect of NaOH molarity on the rate of water absorption

4.2.2.5 Density (molarity)

Table 4.6 shows 28-day densities with the respective NaOH molarity of the mixes. The densities ranged from 1710 to 1754 kg/m^3 for molarities of 8 to 16 using POCS. Based on the density results, it can be observed that there is a direct relationship between the density and the molarity as the density increases with the specimens of higher molar concentrations. It is worth mentioning that in order to increase the concentration of the NaOH solution, more powder should be dissolved in the water, which leads to an increase in the density of the solution. As a result, the increased density of the AAS is one of the causes of the rise in the density of the mixes with higher NaOH concentrations.

On the other hand, the control mix, which uses MS with a molarity of 10, produced a 28-day density of 2077 kg/m^3. The comparison between GP-1 and GP-3 demonstrates the reduction of the density of about 17% due to the whole replacement of POCS instead of conventional sand. Lightweight concrete could be significantly effective in reducing the overall load of the structures (Bremner & Eng, 2001).

Table 4.6: The 28-day density with respective molarity

Mix ID	Molarity	Sand	28-Day density (kg/m^3)
GP-1	10	MS	2076.8
GP-2	8	POCS	1710.4
GP-3	10	POCS	1720.1
GP-4	12	POCS	1719.2
GP-5	14	POCS	1750.6
GP-6	16	POCS	1753.7

4.2.3 Effect of curing condition (Oven vs. ambient) of geopolymer mortar

Mechanical properties and durability aspects of the geopolymers are greatly affected by the curing condition (De Vargas et al., 2011). Alkali-activated materials are mostly cured at 40-80°C for 4-48 hours (Palomo et al., 1999; Temuujin et al., 2009). This curing needs a tremendous amount of energy, and it is diminishing the feasibility of utilizing the geopolymers in a wide range of applications. On the brighter side, the incorporation of GGBS in geopolymers enhances the strength development at the early stages due to the presence of high calcium (Lee et al., 2019). In the current study, the role of GGBS in the strength development under oven and ambient curing conditions was investigated. The oven curing condition was done in a curing chamber with a temperature of about 65°C for 24 hours compared with ambient curing condition of 27-32°C with a relative humidity of about 60-70%.

4.2.3.1 Compressive strength (curing)

The strength development of the oven-cured mixes at the ages of 1-, 7-, and 28-day is shown in Figure 4.20 It can be observed that an increase in the GGBS content from 10-30% enhanced the compressive strength to peak at 50 MPa (70% increase compared to the control mix). However, the addition of GGBS beyond 30% had a negative impact as there was a reduction of the strength as depicted.

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The comparison between GP-1 and GP-9 shows that FA-GGBS-POCS geopolymer mortar can be produced with approximately the same strength as that of the MS geopolymer mortar in which only FA was used as the precursor.

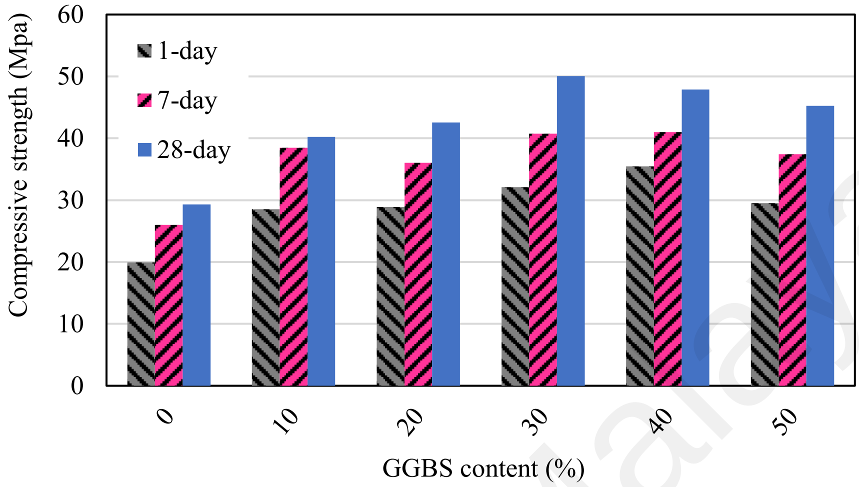

Figure 4.20: Compressive strength of the oven-cured specimens

In contrast, the ambient-cured specimens showed higher strength with higher quantities of GGBS. Referring to Figure 4.21, when GGBS content was increased from 10 to 50%, the strength was improved, and the 28-day compressive strength was found 53 MPa with 50% of replacement. It was reported that the GGBS content has a direct relationship with compressive strength (Soutsos et al., 2016). One of the main reasons could be related to the reaction heat and the hydraulic activity generated from GGBS, which is higher compared to that of FA (Qiang et al., 2016), that leads to higher compressive strength.

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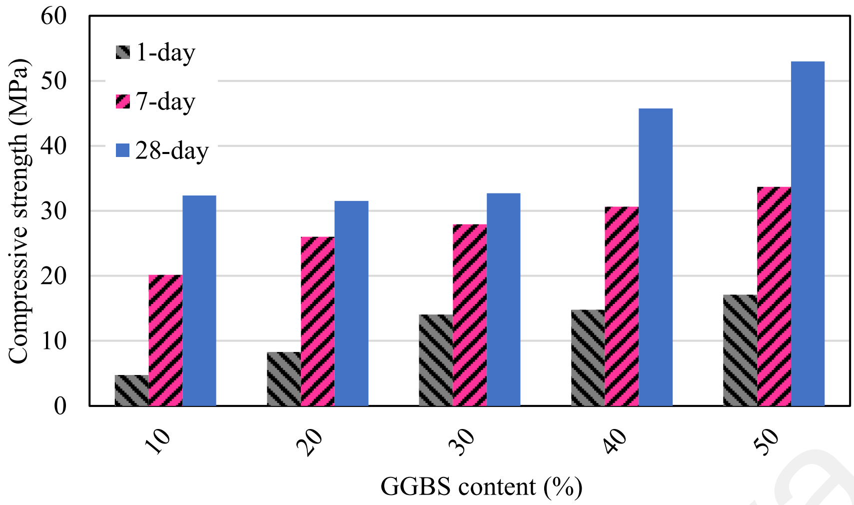

Figure 4.21: Compressive strength of the ambient-cured specimens

Figure 4.22 compares the long-term compressive strengths of the control mix (GP-1) and the ambient-cured mix with 50% of GGBS (GP-16), which acquired the highest 28-day strength. The comparison shows different trends as the compressive strength of the control mix tends to increase with time while the other experienced a reduction of about 14% from the age of 28 to 180 days. Reduction in the strength from 28 to 90 days for mixes with 20% GGBS was reported by Ismail et al. (2013). The long-term strength reduction of the FA-GGBS mixes could primarily be ascribed to the increased formation of C-A-S-H gel with a more porous structure instead of C-S-H gel (Ismail et al., 2013).

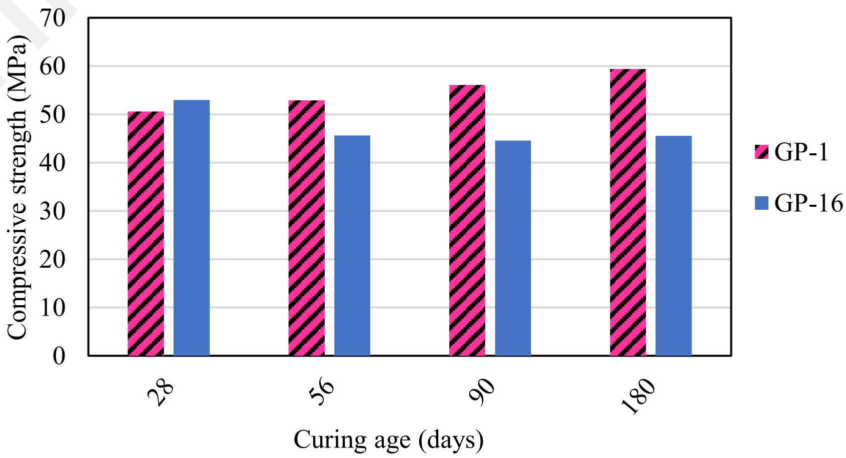

Figure 4.22: Long-term compressive strength

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4.2.3.2 Strength development (curing)

The comparison of strength development is shown in Figure 4.23 as the percentage of 1- to 28-day strength for ambient and oven-cured specimens. Due to the accelerated geopolymerization by the heat generated in the oven, the specimens developed considerably higher strengths within the first 24 hours. The 1-day strength development of the ambient-cured specimens had an upward trend with the increase in GGBS content of up to 30%. For higher GGBS percentages of 40% and 50%, there was a drop in the strength development in which about 32% of the 28-day strength was produced in the first 24 hours. One of the major issues of the FA-based geopolymers is that the strength development requires heat curing (Kaur et al., 2018b), and this could be negated by partial incorporation of GGBS. The incorporation of GGBS as a binder in geopolymer mixes could elevate the strength development to up to 43% in the first 24 hours without the need for oven curing while using FA as the sole binder would not develop any strength without heat curing in the same period.

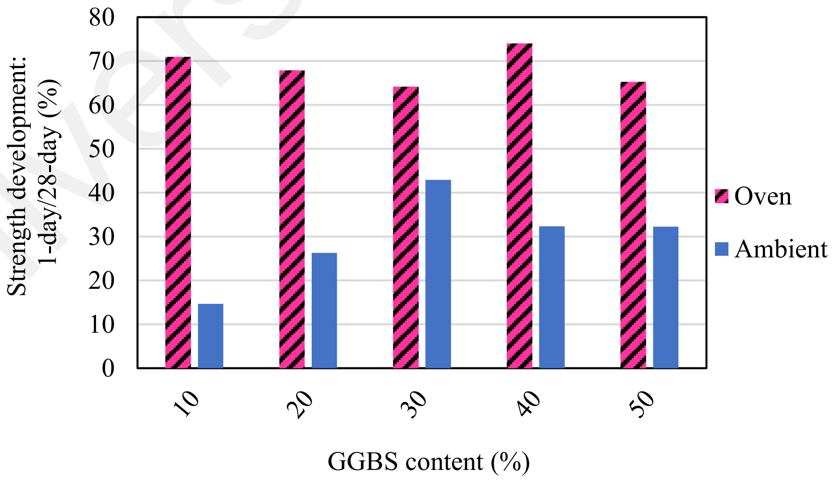

Figure 4.23: Comparison of strength development as the ratio of 1-, and 28-day for ambient and oven-cured specimens

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4.2.3.3 Ultrasonic Pulse Velocity (curing)

The 28-day UPV results of the oven and ambient-cured mixes containing 10-50% of GGBS are demonstrated in Figure 4.24, and Figure 4.25, respectively. The trend shows an improvement in the UPV values with the increase in GGBS contents, and ambient-cured specimens produced higher velocities than the oven-cured ones. The incorporation of the GGBS in the mixes contributed to the development of the geopolymerization as the UPV result increased substantially compared to the mixes without GGBS. The FA based geopolymer recorded 2.5 km/s, but the replacement of 10-50% of FA by GGBS resulted in UPV values of 2.75-3.36 and 2.79-3.71 km/s for the oven and ambient-cured specimens, respectively. According to the researchers (Hwang et al., 2012), UPV value of 3.71 km/s produced by the mix with 50% GGBS could be categorized as “excellent”.

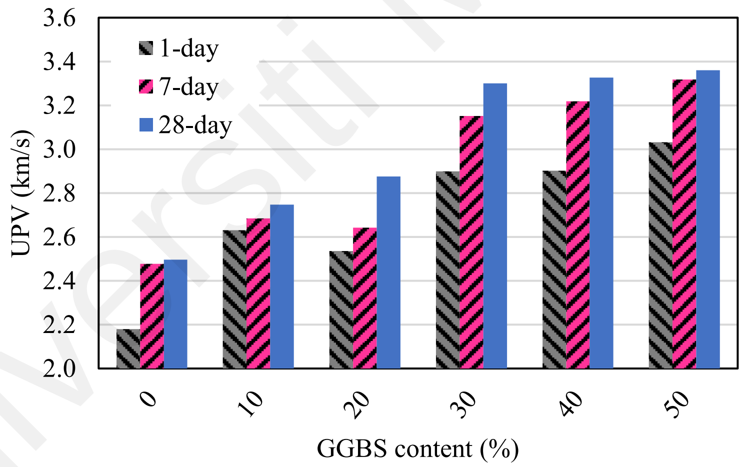

Figure 4.24: Ultrasonic pulse velocity of oven cured mixes

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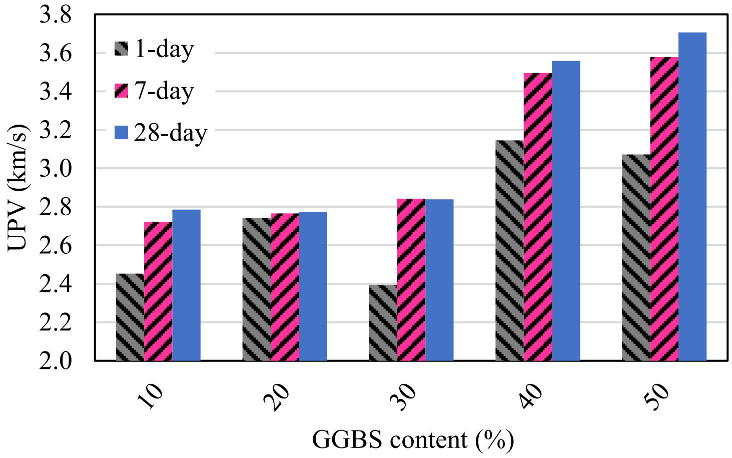

Figure 4.25: Ultrasonic pulse velocity of ambient cured mixes

4.2.3.4 Water absorption (curing)

It was reported by Ghosh (2012) that concrete mixes with higher mechanical properties would result in lower water absorption. The water absorption of both oven (Figure 4.26) and ambient-cured (Figure 4.27) specimens decreased gradually with the increase in GGBS contents. However, the ambient-cured specimens experienced lower water absorption rates, and the mix with 50% of GGBS had the lowest value of 9.7% among all the mixes. The water absorption of alkali-activated concrete was reported to be lower than that of the ordinary Portland cement concrete specimens for a similar binder content (Bernal et al., 2011a), and this was attributed (Bernal et al., 2011b) to the inclusion of finer particles of GGBS that filled the pores that diminishes the penetration of water. The ambient-cured mixes with 30 - 50% of GGBS had a water absorption of less than 10%.

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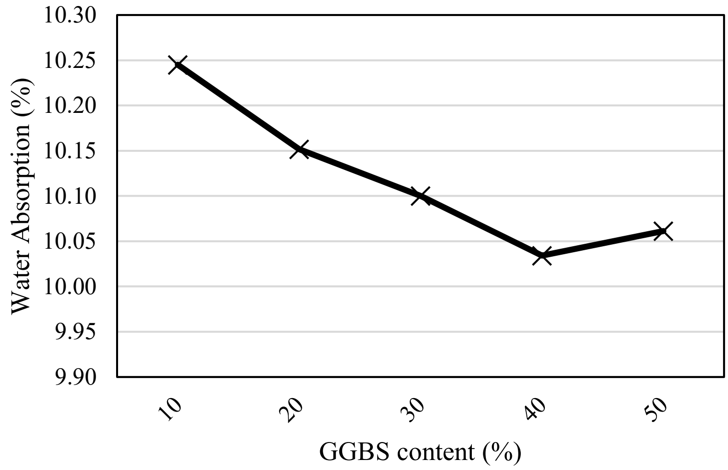

Figure 4.26: Effect of GGBS content on water absorption of oven-cured specimens

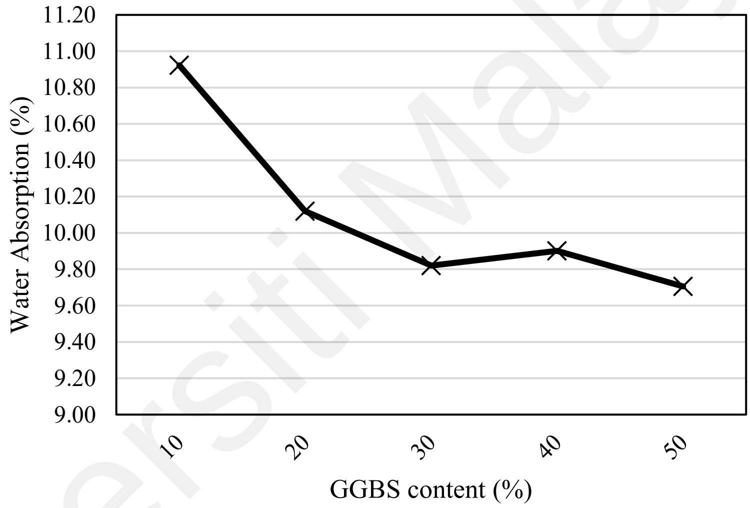

Figure 4.27: Effect of GGBS content on water absorption of ambient-cured specimens

4.2.3.5 Rate of water absorption (curing)

The rate of water absorption test was carried out to investigate the effect of GGBS content on the sorptivity of the mortars for both oven (Figure 4.28) and ambient-cured (Figure 4.29) specimens. Both the curing methods showed almost similar trends; the rate of absorption decreased with the increase in the GGBS contents. Nevertheless, the oven-cured specimens with 50% of GGBS experienced an increase in the absorption rate, and this could be due to excess heat generated by the GGBS and the heat from the oven, which resulted in the evaporation of the water. Thus, the water absorption of the mix was increased.

The incorporation of GGBS could cause secondary pozzolanic reactions, and this could contribute to the filling of pore structures; this enables the concrete to reduce the possibility of chemical attack and penetration (Divsholi et al., 2014). This feature enables the mixes with GGBS to have better resistance towards water absorption.

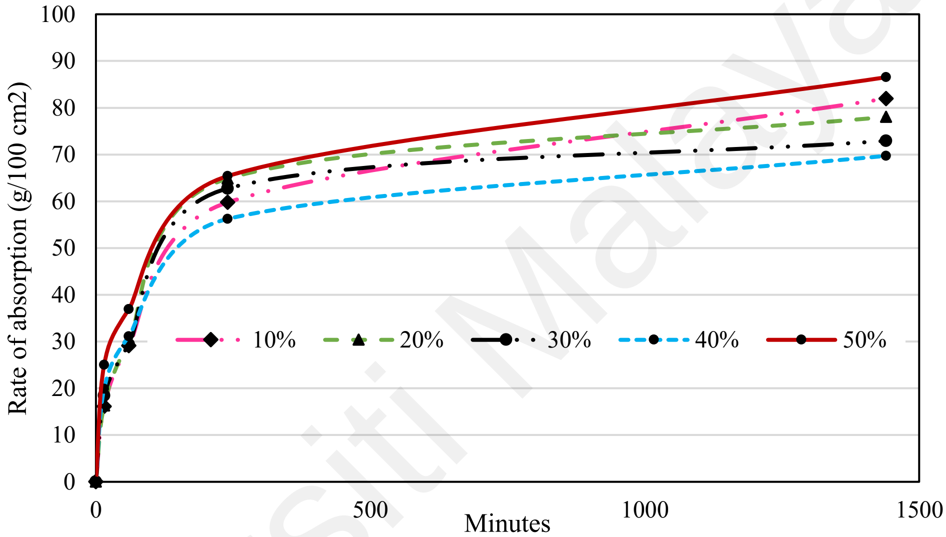

Figure 4.28: Effect of GGBS content on the rate of water absorption of oven-cured specimens

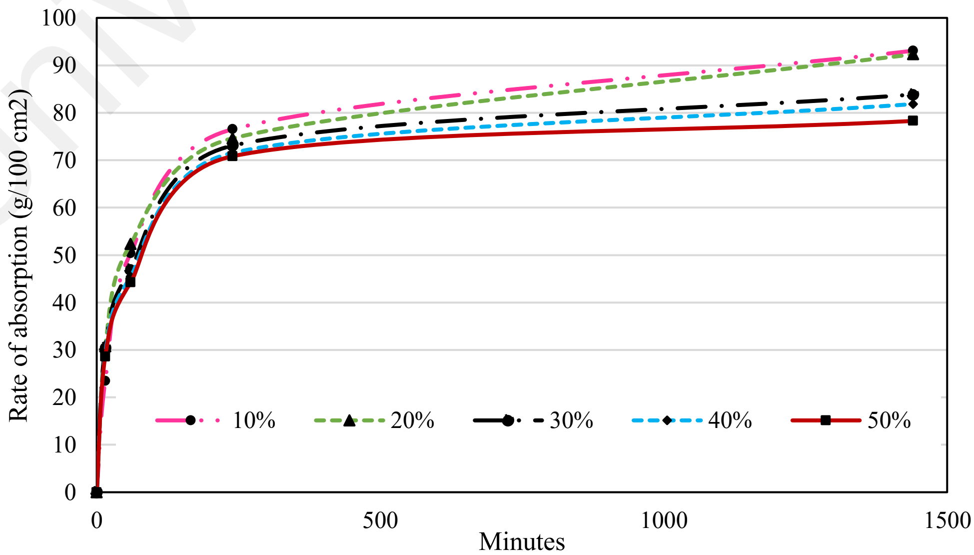

Figure 4.29: Effect of GGBS content on the rate of water absorption of ambient-cured specimens

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4.2.3.6 Density (curing)

Figure 4.30 shows the 28-day density of the specimens for different percentages of GGBS for the oven and ambient-cured conditions. As known, the specific gravity of the GGBS is higher, and as a result, the replacement of FA with GGBS increased the final density of the specimens. The density of all the mixes fell within the range of 1754 -1842 kg/m^{3,} and according to the European Standard of EN 206-1 (European standard, 2000), the oven-dry density of 900-2000 kg/m^3 is categorized as lightweight concrete (LWC). Remarkably, most types of LWCs utilize recycled and industrial waste materials, which could ideally fall under sustainable and energy-efficient materials category (Chung et al., 2019).

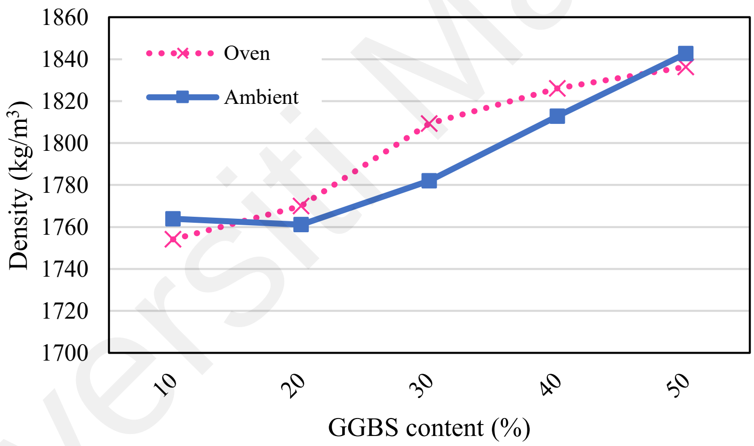

Figure 4.30: Effect of GGBS on the 28-day density of the specimens

4.2.3.7 Setting time (curing)

One of the governing factors for geopolymers is the setting time since the matters of transportation, compaction, and the pouring of the concrete substantially depend on the setting time of the mix. The humidity of the lab was observed 50% with 23°C of temperature for the ambient mixes and a 65°C of temperature for the oven mixes. The ambient-cured mixes with class-F fly ash as the binder have a setting time of at least 24 hours (Hardjito et al., 2004). With the incorporation of GGBS, however, the setting time reduced (Rafeet et al., 2017). The setting time of the FA-GGBS geopolymer mortars for both oven, and ambient-cured specimens was measured (Mixes GP-7 to GP-16) and depicted in Figure 4.31. The increase in GGBS involvement caused a reduction in the setting time for both curing methods as it was also verified in the literature (Elyamany et al., 2018; Nath & Sarker, 2014; Rafeet et al., 2017). The setting time of the oven-cured mixes ranged from 44 to 23 minutes for the GGBS involvement of 10 to 50%, while for the ambient-cured mixes it ranged from 77 to 29 minutes, respectively.

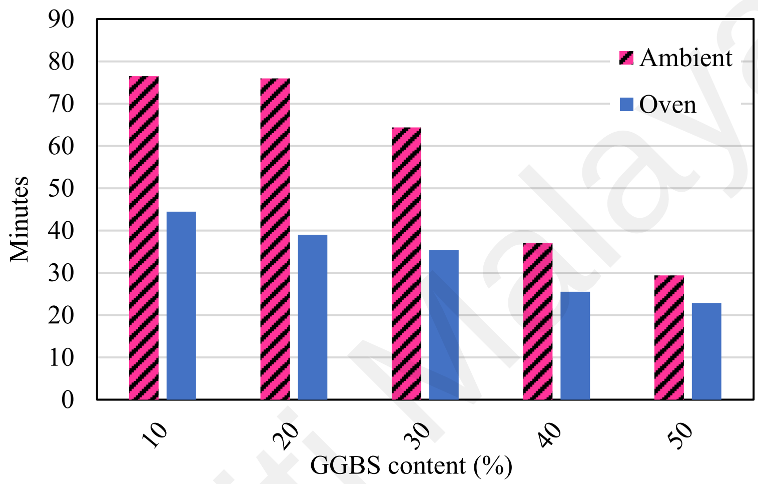

Figure 4.31: Setting time of FA/GGBS mixes

4.2.4 Chemical resistivity of geopolymer mortar

The durability aspect of the concrete derivatives can demonstrate the resistance of the product towards chemical attacks and weathering conditions. In order to examine the chemical resistance, 50-mm cubes were cast and cured for 28 days prior to immersing those in hydrochloride acid (HCl) and magnesium sulfate ($MgSO_4$). Three mixes were chosen for this purpose; GP-1 represents the control geopolymer mortar that utilized 100% FA as binder and MS as the fine aggregate, while the mix GP-16 contains an equal amount of FA-GGBS (50:50) as binders and POCS as the fine aggregate which acquired the highest mechanical properties. Lastly, for comparison of the effect of these chemicals on geopolymer mortar, a cement-based mixture with the same paste volume was prepared and tested along with the other two mixes. These three mixes represent MS and POCS geopolymer mortars along with cement mortar. Three cubes were used for each period of testing, and the average values of the strength and weight loss were taken and reported.

4.2.4.1 Acid resistance

Figure 4.32 shows the specimens after 28 days of submersion in the acid solution.

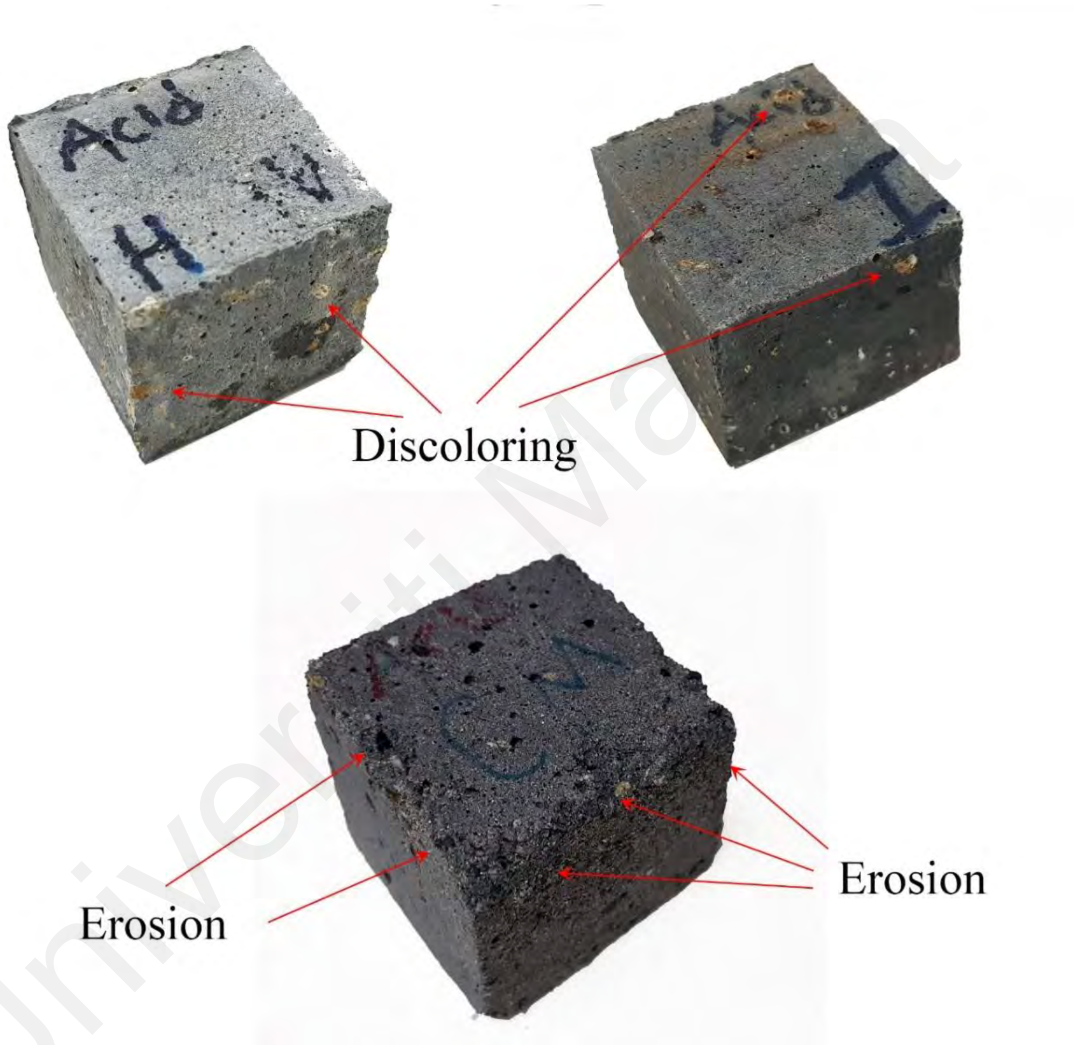

Figure 4.32: Physical inspection after 28 days of immersion in HCl a) FA-MS geopolymer mortar, b) FA-GGBS-POCS geopolymer mortar, c) cement-MS mortar

It is interesting to note from Figure 4.33 that the geopolymer specimens had no signs of erosion and physical damages to the surface even after 180 days of exposure to acid. Slight discolouring, however, was observed on the surface for both mixes that could be washed with a thorough cleansing. Nevertheless, the cement mortar suffered from erosions, and the edges were seen severely damaged.

a) b)

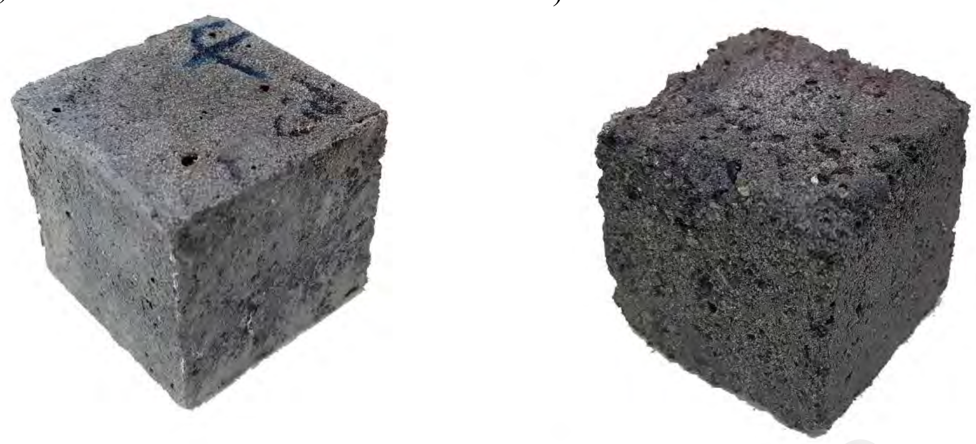

Figure 4.33: Physical inspection after 180 days of immersion in HCl a) FA/GGBS/POCS geopolymer mortar b) cement/MS mortar

The variation of the compressive strength of the specimens that were immersed in acid is shown in Figure 4.34. The POCS and MS geopolymer mortars demonstrated a satisfactory resistivity especially during the first 90 days of immersion; the residual strength of the specimens was found more than 60% of their initial strength; in contrast, the cement mortar showed a very steep decline in strength as almost 80% of strength was lost. Overall, the POCS geopolymer mortar exhibited relatively good resistance close to that of the control FA-MS geopolymer mortar.

Figure 4.35 shows the deteriorating effect of the cement mortar after the 180-day compressive strength test; the discolouring effect along with spalling of mortar even in the interior parts of the cement mortar is very much evident. However, the FA-MS and FA-GGBS-POCS based mortars showed excellent resistance and the interior surfaces had no deteriorating effect due to acid. The presence of the alumino-silicate compound in geopolymer mortar has a better resistance against acid attack compared to the calcium hydroxide compound in cement mortar (Albitar et al., 2017; Chindaprasirt & Rattanasak, 2017) and the fragility of the cement mortar after 180 days in the HCl solution was beyond doubt (Figure 4.35).

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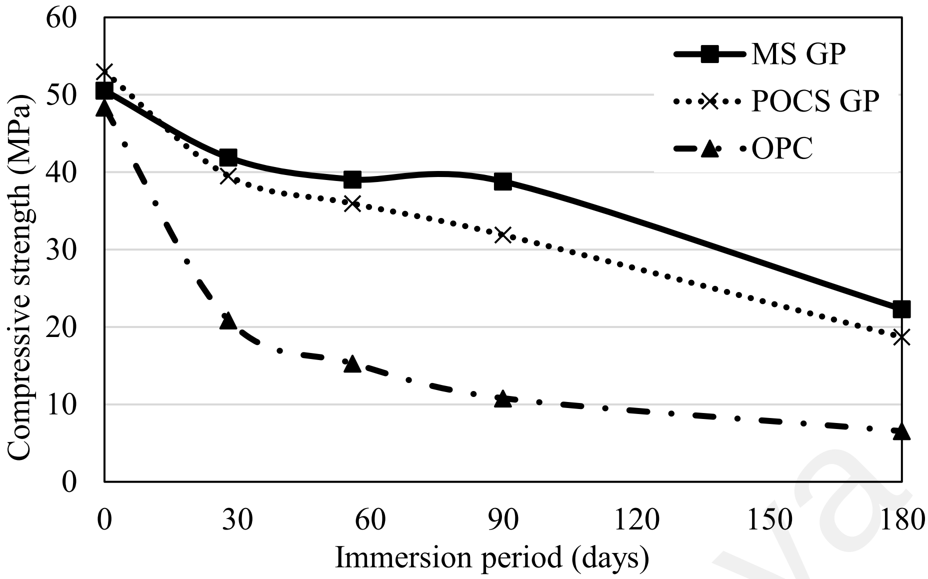

Figure 4.34: compressive strength of the samples after immersion in HCl

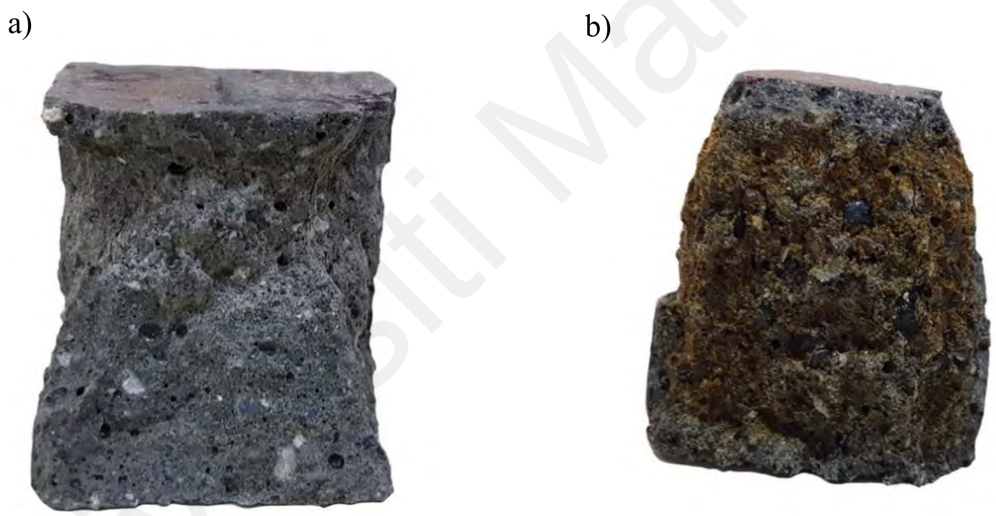

Figure 4.35: Compression test after 180 days of immersion in HCl a) FA/MS geopolymer mortar b) cement/MS mortar

The effect of acid on the change in dimensions in the geopolymer mortars was not observed. Conversely, the cement mortar specimens had an average reduction of 0.05 to 0.20 mm, and the effect of acid on the corners, and the edges was evident from the specimen, as depicted in Figure 4.33. The weight loss of the specimens during the immersion period is shown in Figure 4.36 as a percentage to their initial weight. The geopolymer specimens of mixes GP-1 (MS) and GP-16 (POCS) experienced weight losses of 2.62 and 4.12% after 180 days, respectively, and by observation, it is clear that most of the weight loss happened between 28 and 90 days for the geopolymer mortars. On the contrary, the cement mortar had shown a sharp increase in its weight loss amounting to 15.4% during the whole period of testing, and this could be attributed to the presence of calcium hydroxide (Kim et al., 2014).

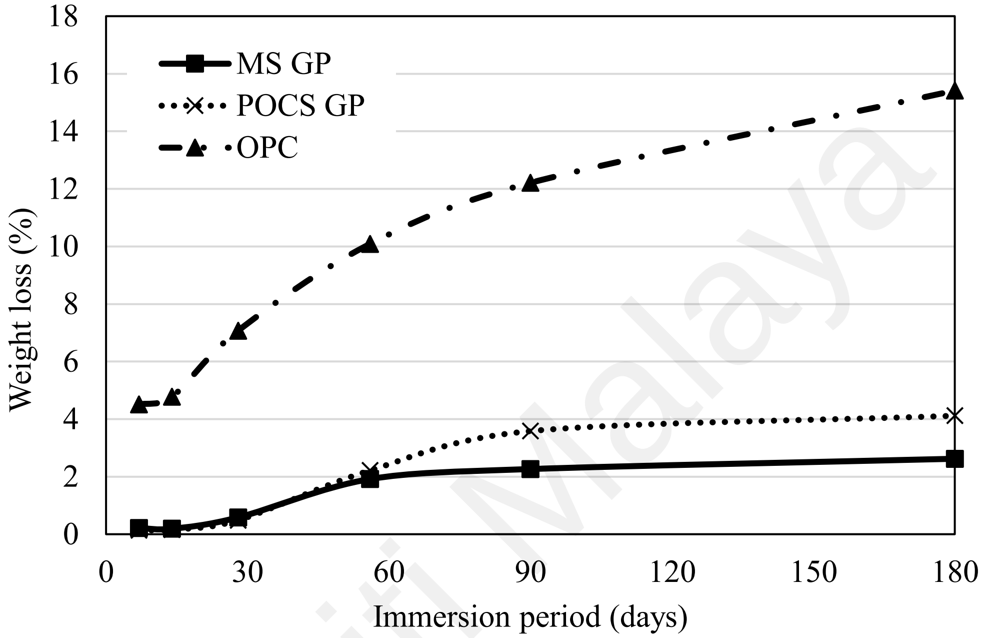

Figure 4.36: Weight loss of samples soaked in HCl

4.2.4.2 Sulfate resistance

Sulfate is one of the main causes of concrete corrosion and deterioration that forms a new reaction with the sulfate ions in the pore structure of the concrete. Figure 4.37 shows the residual compressive strengths, and as observed, all the mixes show a reduction in their strength mainly after 56 days of immersion. Geopolymer mortars outperformed the cement mortar in resisting against the sulfate.

The control FA-MS geopolymer mortar had the lowest reduction in the strength of about 20%, followed by 30% and 40% reduction for the POCS geopolymer and cement mortar, respectively; the residual compressive strength of POCS geopolymer was observed 36.7 MPa after 180 days of immersion. It can be understood that geopolymer mortar has a better resistance toward $MgSO_4$ owing to its denser matrix than the cement.

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However, comparison between the MS and POCS geopolymer mortars shows that the sulfate solution has penetrated to the inner parts of the POCS mortar more intensively compared to that of the MS mortar. Therefore, the POCS mortar experienced a higher reduction over the period.

The presence of C-S-H in the cement mortar reacts with the sulfate, resulting in ettringite, whereas gypsum and brucite, along with the ettringite are the products of the geopolymer mortar with the $MgSO_4$ solution (Sukmak et al., 2015). Interestingly, the majority of the strength loss of the cement mortar specimens occurred between 90 and 180 days since the trend of its reduction had an almost same steep to the geopolymer mixes during the first three months.

The appearance of the specimens of all the three mixes was observed almost intact, and the weight of the cube specimens was not reduced, and, in some cases, a slight increase was noticed over the period of the immersion.

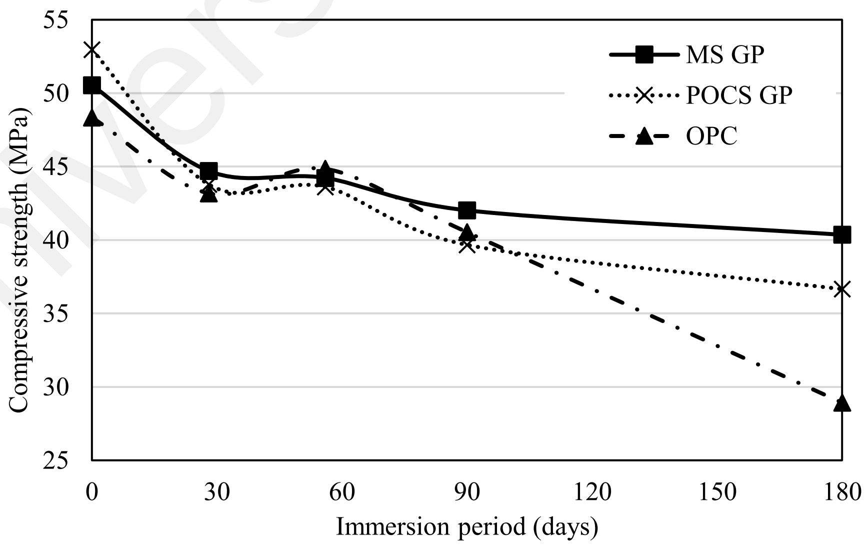

Figure 4.37: Compressive strength of the cubes soaked in the magnesium sulfate

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4.2.5 XRD analysis of geopolymer mortar

XRD analysis was conducted to study the microstructural behaviour of the mixtures using pan analytical XRD equipment. Figure 4.38 shows the diffraction patterns of the raw materials of FA and GGBS. Both binders possess a hump showing an amorphous phase of the materials. However, the hump is broader for FA between 16 and 35 compared to the 23 to 38 of 2θ degrees for GGBS. Quartz and Mullite are dominant in FA, which confirms a higher amount of silica, while GGBS shows the presence of Calcite and Periclase of higher magnesia and calcium oxide contents.

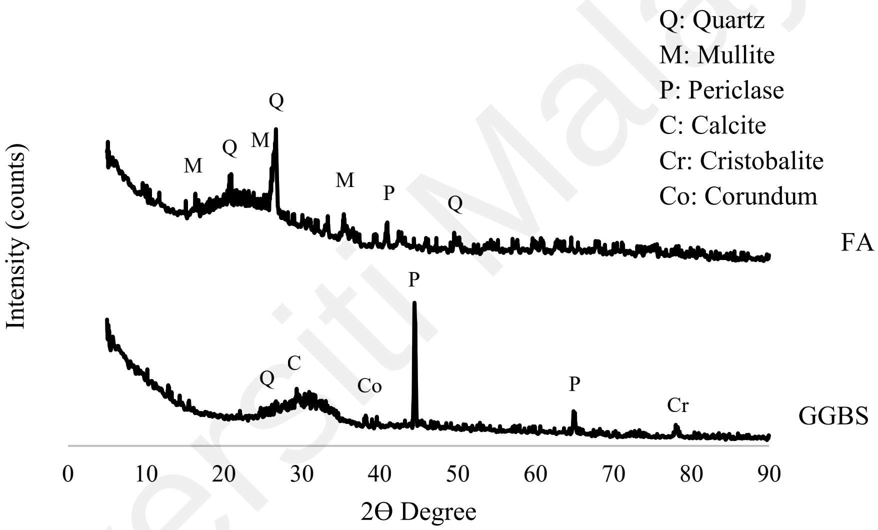

Figure 4.38: XRD analysis of FA and GGBS

In order to investigate the effect of GGBS and curing in the geopolymerization process, selected geopolymer pastes, as shown in Table 4.7 were prepared and analyzed after 28 days.

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Table 4.7: Geopolymer pastes prepared for XRD analysis

Mix ID	Binder	Curing
1, 3	FA	65°c for 24 hours
9	70%FA - 30% GGBS	65°c for 24 hours
14	70%FA - 30% GGBS	Ambient
11	50%FA - 50% GGBS	65°c for 24 hours
16	50%FA - 50% GGBS	Ambient

Figure 4.39 shows the X-ray diffraction of geopolymer pastes with 2θ being between 5 and 90 degrees. Overall, the comparison between ambient and oven-cured mixes show identical peaks with minor differences. This resemblance confirms the similar geopolymerization of the mixes of FA-GGBS at ambient and oven-cured conditions, and a similar outcome was reported for FA-POFA geopolymer mortar (Ranjbar et al., 2014). It is understood that most crystalline phases of the Quartz and Mullite present in raw FA, were later dissolved and took part in the reaction process as seen from the diminished peaks in the paste. At 26.2 and 40.8 degrees, the presence of Mullite in the mix of 100% FA shows resolved in lower FA quantities. This implies that the excess phases of the Mullite were slightly inactive in the FA paste.

Conversely, new phases of Periclase were recognized as a result of geopolymerization in which more peaks were identified in the mixes with higher percentages of GGBS. Calcite was another product of the process which nevertheless was most notable in the ambient-cured mixes incorporating GGBS. This could be ascribed to the high values of CaO (Wongsa et al., 2017) present in the GGBS (Table 3.1) and, consequently, might have contributed to the development of strength in the ambient-cured mixes containing GGBS (Sharmin et al., 2017; Yusuf et al., 2014).

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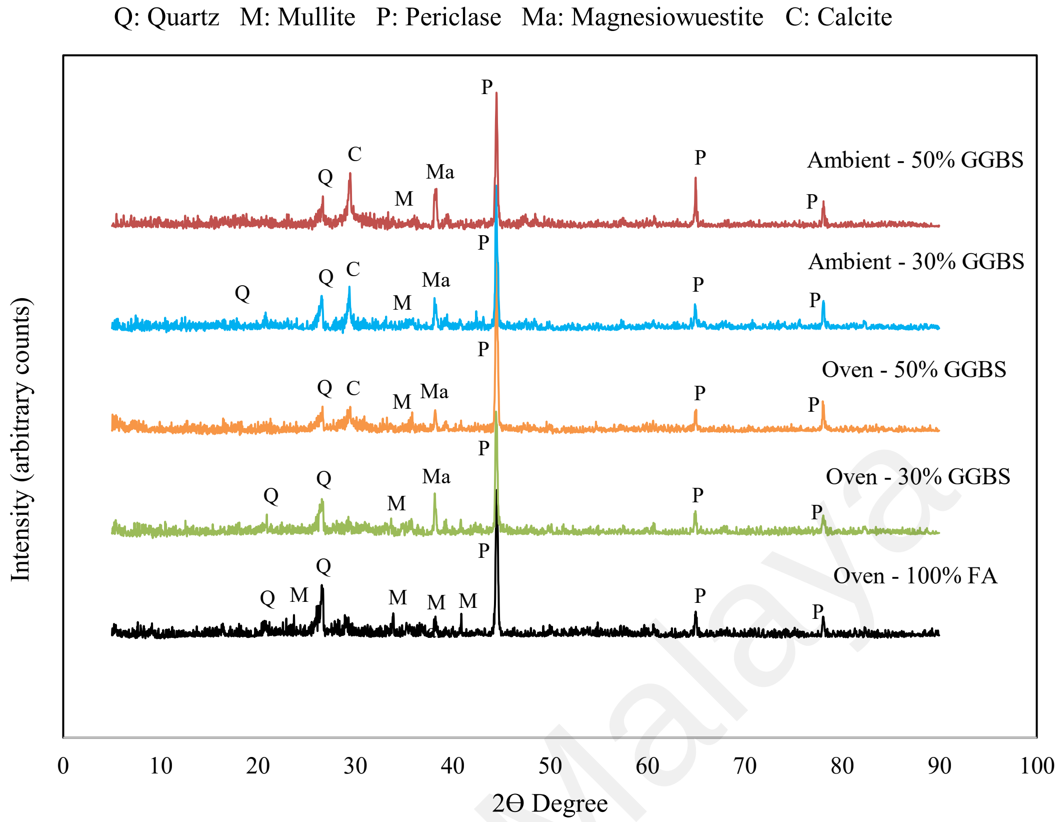
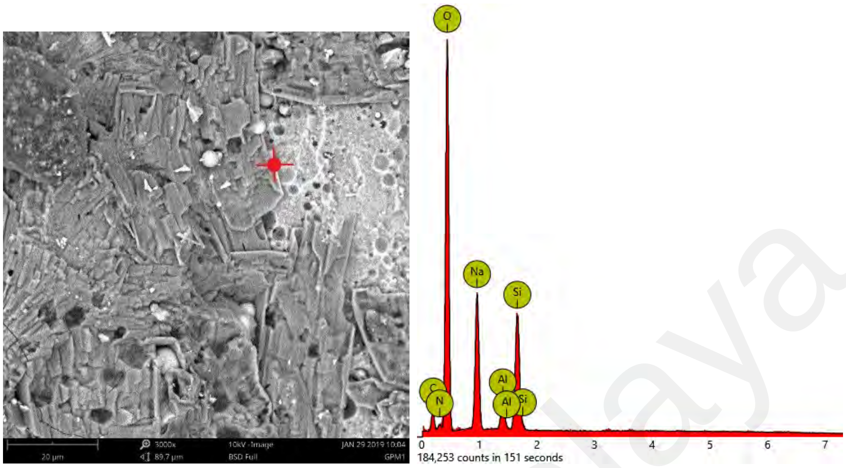


Figure 4.39: XRD analysis of geopolymer pastes

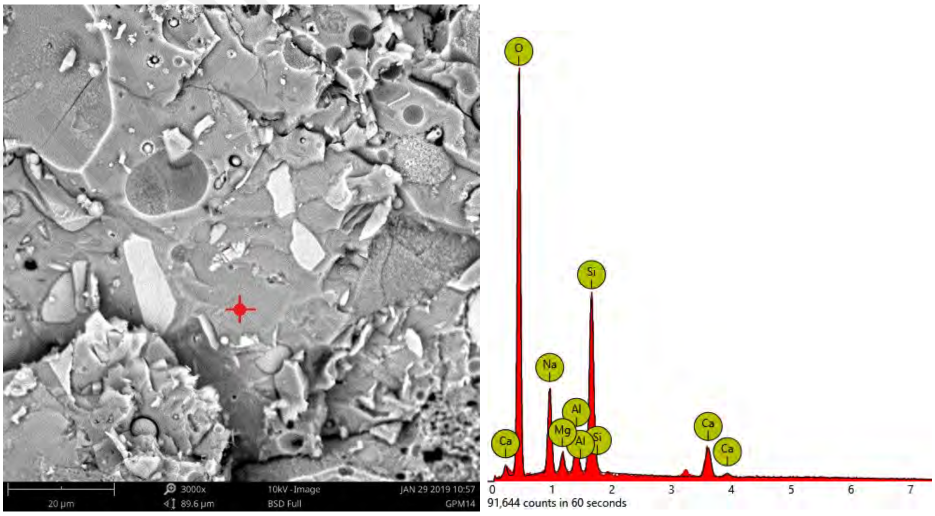
4.2.6 Energy dispersive x-ray (EDX) analyse of geopolymer mortar

The EDX analysis was performed on the mixes GP-1 and GP-16 to characterize the chemical processes involved in the geopolymerization. Figure 4.40 and Figure 4.41 show SEM images along with the elemental concentrations of the chosen spots. According to the identified components, the typical elements of GP-1 and GP-16 are O, Na, Si, Al, and both mixes show the existence of sodium, and this is attributed to the activator's solution where it is the main element (Kourti et al., 2010). Ca and Mg were also detected in mix GP-16 as these are present in GGBS. Based on the concentrations of the elements, the Si/Al ratios are 4.2 and 4.5 for GP-1 and GP-16, respectively. A previous study (He et al., 2016) revealed that an increase in Si/Al ratio results in improved mechanical properties since more Si-O-Si bonds will occur. On the other hand, high calcium and silicate with low aluminium lead to the formation of calcium-silicate-hydrate (C-S-H) (Ariffin et al., 2013; Mijarsh et al., 2014) while higher amounts of aluminium with the same combination will create C-A-S-H products which are reported to have a dense structure, establishing an excellent mechanical outcome (Puertas et al., 2011). Thus, the enhanced strength of the mix GP-16 could be contributed by Si-O-Si and C-A-S-H bonds.



Element	Oxygen	Sodium	Silicon	Carbon	Aluminium	Nitrogen
Atomic conc.	60.56	12.16	8.2	13.49	2.03	3.56
Weight conc.	55.51	16.01	13.19	9.28	3.14	2.86

Figure 4.40: EDX spectrum with elemental concentrations (GP1)



Element	Oxygen	Silicon	Calcium	Sodium	Aluminium	Magnesium
Atomic Conc.	70.16	12.73	4.62	7.94	2.95	1.59
Weight Conc.	57.09	18.19	9.42	9.29	4.05	1.97

Figure 4.41: EDX spectrum with elemental concentrations (GP16)

4.3 Effect of POCS grading on the performance of the mortar

The majority of the weight of the mortar is attributable to the sand, and, as a result, the importance of sand cannot be neglected as it directly influences the fresh and hardened properties of the mortar. One of the most vital factors in determining the quality of sand is the grading of the sand. Thus, the effect of POCS grading on different properties of cement and geopolymer mortars was investigated and compared with the control mixes featuring MS. Relative proportions of POCS with sizes of 0.15 - 4.75, 0.15 - 2.36, 0.15 - 1.18, and 0.15 - 0.60 mm were used to form four categories of gradings. The POCS with the grade of 0.15 - 4.75 was used with the same condition as collected without any changes to the grading. However, for the other mixes, a special grading was applied, as suggested by Karim et al. (2016), which was graded 0.15 – 2.36 mm and then modified to get another two gradings of 0.15 – 1.18 and 0.15 – 0.60 mm, as shown in Table 4.8. The purpose of this grading was to observe the effect of the POCS fineness on the properties of the mortar.

Table 4.8: Sand grading details

ID	Type		Range	Grading groups (%)				
				2.36-4.75	1.18-2.36	0.60-1.18	0.30-0.60	0.15-0.30
G-1	Cement mortar	MS	0.15 – 4.75	25	28	25	10	7
G-2		POCS	0.15 – 4.75	32	23	19	15	7
G-3		POCS	0.15 – 2.36	-	25	35	20	20
G-4		POCS	0.15 – 1.18	-	0	60	20	20
G-5		POCS	0.15 – 0.60	-	0	0	80	20
G-6	GP mortar	MS	0.15 – 4.75	25	28	25	10	7
G-7		POCS	0.15 – 4.75	32	23	19	15	7
G-8		POCS	0.15 – 2.36	-	25	35	20	20
G-9		POCS	0.15 – 1.18	-	0	60	20	20
G-10		POCS	0.15 – 0.60	-	0	0	80	20

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Figure 4.42 shows the sieve analysis of different POCS gradings along with the upper and lower limits of the European standard (European standard EN 13139, 2002). As can be observed from the graph, POCS 0.15 - 4.75 is not in the proposed range, while all the other graded groups are in line with the mentioned standard.

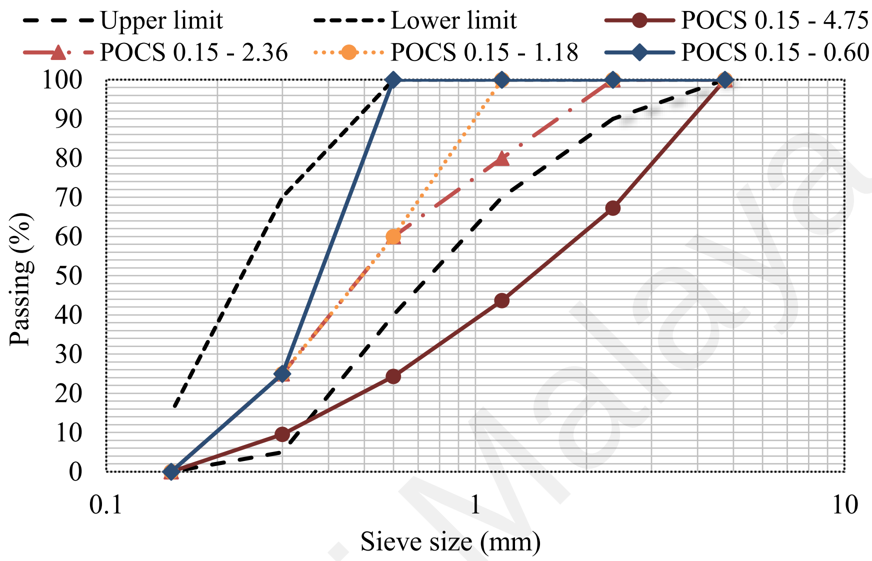

Figure 4.42: Sieve analysis on the POCS gradings

The physical properties of the POCS gradings and MS are shown in Table 4.9. It is noted from the table that the specific gravity of the POCS is much lower than that of conventional sand. This is due to the porous nature of the POCS that makes it lightweight and water absorbent. Decreasing the particle sizes of the POCS resulted in a substantial increase in the specific gravity of 1.94 for the POCS graded 0.15 – 4.75 to reach 2.11 with a grade of 0.15 - 0.60. Moreover, the water absorption of the finer POCS sand was found to be higher (as shown in Table 4.9), which could be ascribed to the increased surface area of the finer particles.

Table 4.9: Physical properties of POC gradings

Grading	Fineness Modulus	Specific gravity	Water absorption (%)
MS 0.15 - 4.75	3.43	2.65	0.24
POCS 0.15 – 4.75	3.55	1.94	3.30
POCS 0.15 – 2.36	2.35	1.97	3.31
POCS 0.15 – 1.18	2.15	2.01	4.75
POCS 0.15 – 0.60	1.75	2.11	5.69

The binders used in this study were ordinary Portland cement (OPC), and equally distributed fly ash (FA) and ground granulated blast-furnace slag (GGBS) for the conventional and geopolymer mortars.

4.3.1 Grading of POC in cement and geopolymer mortars

A water to cement (w/c) ratio of 0.485 was used for the control cement mortar using the MS, which was adopted from ASTM C109 (ASTM International, 2016a). For the cement mortars using POCS, however, a higher w/c ratio of 0.60 was used to compensate for the higher water absorption rate of POCS, as advised by previous researchers (Kanadasan et al., 2018; Ramappa Ramesh et al., 2018).

To increase the flowability and consistency of the geopolymer mixes, additional free water was added to the mixes. The amount of free water for the MS and POCS geopolymer mixes was 10% and 17% of the binder's weight, respectively.

The replacement of the sand was based on the specific gravities of MS and POCS to produce the same paste volume for both the cement and the geopolymer mortars. Thus, the same paste volume was applied for all the mix designs to have a more efficient comparison between the mixes. The mix proportions of the cement and geopolymer mortars are given in Table 4.10 and Table 4.11, respectively.

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Table 4.10: OPC mortar mix proportions - grading

Mix ID	Grading	Sand type	Sand (kg/m^3)	Cement (kg/m^3)	Water (kg/m^3)
G-1	0.15 - 4.75	MS	1368	603	292
G-2	0.15 – 4.75	POCS	1002	527	316
G-3	0.15 – 2.36	POCS	1002	527	316
G-4	0.15 – 1.18	POCS	1002	527	316
G-5	0.15 – 0.60	POCS	1002	527	316

Table 4.11: Geopolymer mortar mix proportions - grading

Mix ID	Grading	Sand type	Sand (kg/m^3)	FA (kg/m^3)	GGBS (kg/m^3)	NaOH (kg/m^3)	Silicate (kg/m^3)	Free water (kg/m^3)
G-6	0.15 - 4.75	MS	1348	298	298	119	179	60
G-7	0.15 – 4.75	POCS	963	282	282	113	169	96
G-8	0.15 – 2.36	POCS	963	282	282	113	169	96
G-9	0.15 – 1.18	POCS	963	282	282	113	169	96
G-10	0.15 – 0.60	POCS	963	282	282	113	169	96

The usage of GGBS as one of the precursors in the geopolymer mixes contributes to the early-stage development of the strength. Thus, GGBS was incorporated to omit the need for oven curing.

4.3.1.1 Flow test of POCS grading

The result of the flow is shown in Figure 4.43. An increase in the finer particles resulted in a reduction in the flowability of the cement and geopolymer mortars. The increased surface area of the particles could be the main culprit for the decreased flow since the lubrication of the sand particles is mainly dependent on their shape (Estephane et al., 2019) and size (Haach et al., 2011). The cement and the geopolymer mortars incorporating POCS produced almost similar flow results for the same sand grading. For the MS cement and geopolymer mortars, the flow table was measured at 55% and 100%,

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respectively. Overall, it could be understood from Figure 4.43 that POCS mortars could produce a flowability of well above 40%.

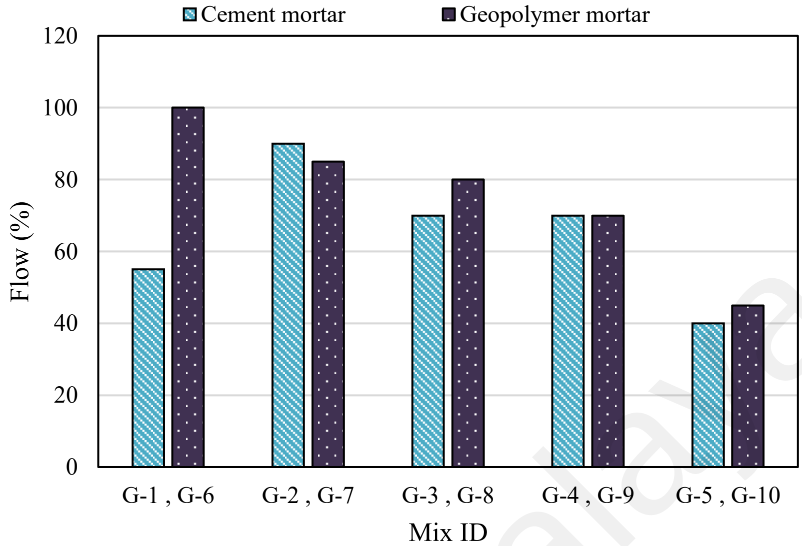

Figure 4.43: The flow table test of cement and geopolymer mortars

4.3.1.2 Compressive strength of POCS grading

The compressive strength of the cement and geopolymer mixes is shown in Figure 4.44 and Figure 4.45, respectively. The cement mixes with both MS and POCS experienced almost constant rise in the strength over the whole period. However, the cement mortar with MS produced a significantly higher 28-day compressive strength of 49.7 MPa compared to the POCS mixes. This difference is largely caused by the difference in the w/c ratios of 0.485 and 0.60 for the MS and POCS mixes, respectively. The porous character of the POCS makes it highly water-absorbent, and higher water content has been reported to have unfavourable effects on the mechanical properties of the mixes (Lotfi-Omran et al., 2019; Piasta & Zarzycki, 2017).

It can be seen from Figure 4.44 that the compressive strength of the POCS cement mortar increased slightly for the mixes with finer sand particles to reach a 180-day strength of 41 MPa for the most fine-grained sand mix. This trend could be due to the better filling feature of the finer particles (Lim et al., 2013a). Furthermore, the smaller sand particles could lead to higher strength materials compared to the larger ones (Guan et al., 2020). The specimens using the larger sand particles possess bigger pores on the surface and the inside since the larger particles are usually less uniform than the small ones. Thus, the mixes using smaller sand particles produced a better compressive strength compared to the bigger sand particles.

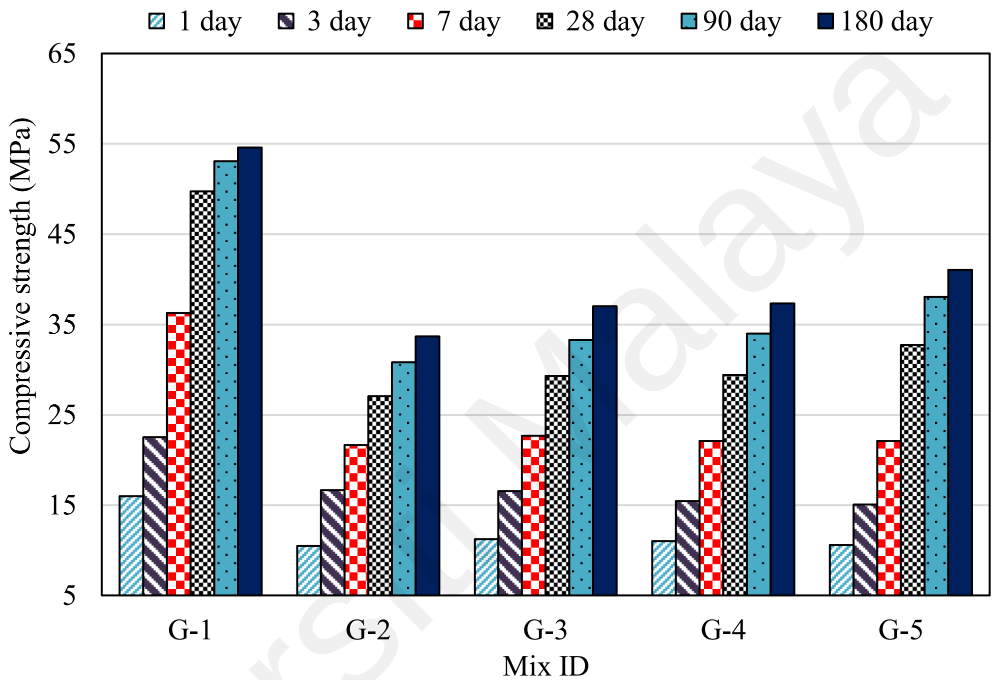

Figure 4.44: Compressive strength of cement mortar specimens

The effect of POCS grading on the geopolymer mixes, however, was less influential as the strength was almost constant with the change in the sand grading, although a gradual rise was observed from POCS grading of 0.15 - 4.75 to 0.15 - 1.18. It is noted from Figure 4.45 that both the MS and POCS geopolymer mortars experienced a reduction in strength between 90 and 180 days of curing. The reduction for the MS mortar was observed around 15%, while for the POCS specimens, it was less than 5%. The mixes utilizing FA and GGBS as binders tended to develop more C-A-S-H gel and comprised a more porous formation compared to the C-S-H gel, which could adversely affect the strength (Ismail et al., 2013). Nevertheless, the 180-day strength of the POCS mortars was recorded at around 60 MPa.

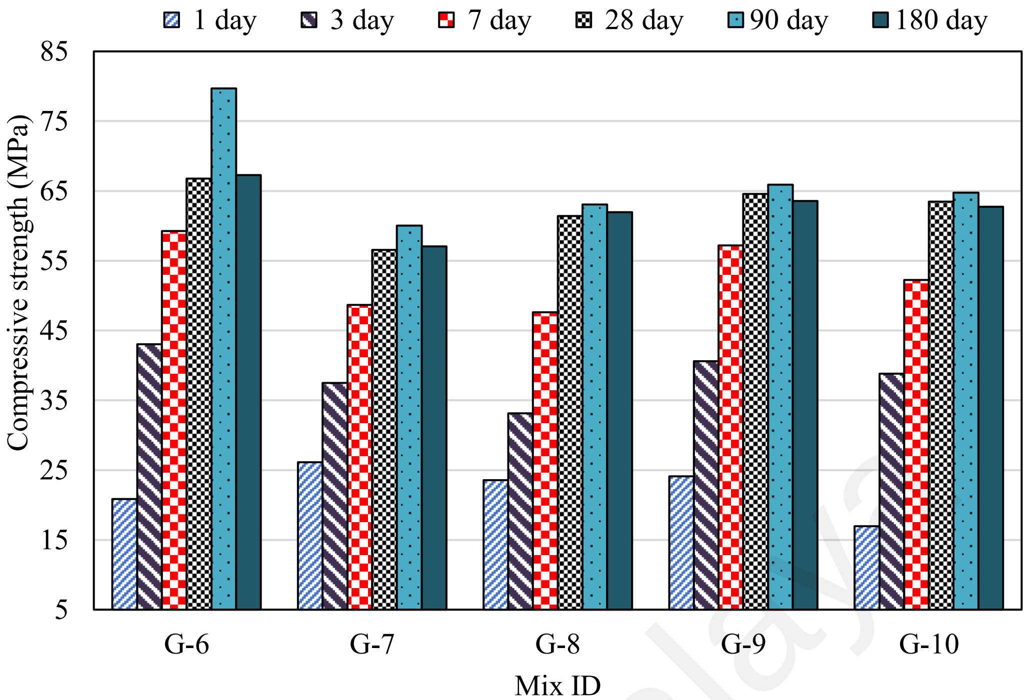

Figure 4.45: Compressive strength of geopolymer mortar specimens

4.3.1.3 Density of POCS grading

Figure 4.46 shows the 28-day density of the cement and geopolymer mortar cube specimens. The highest densities belong to the MS cement and geopolymer mortars with a density of around 2200 kg/m^3, while the POCS specimens of both groups recorded up to 20% lower densities compared to the mixes with MS. This could be attributed to the lower specific gravity and porosities present in POCS. Overall, the densities tend to increase with the utilization of finer sand particles, which is influenced by the packing capability of the particles. It has been reported that the fineness of fine aggregates is considered to be an influential factor in the density and, thus, the properties of the specimens (Venkatarama Reddy & Gupta, 2008). The lower fineness modulus of the sand increases the w/c ratio, leading to lower densities, which, consequently, reduces the strength of the specimens (Venkatarama Reddy & Gupta, 2008).

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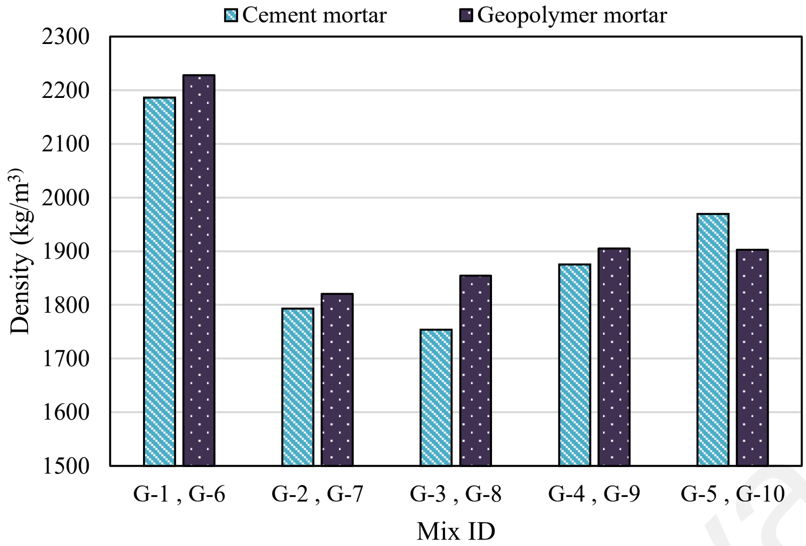

Figure 4.46: The 28-day density of the cube specimens - grading

4.3.1.4 Flexural strength of POCS grading

The particle size of sand significantly affects the flexural strength of the mortar. Using smaller sand particles in the mortar could result in drying shrinkage cracks which reduce the flexural strength of the specimens. The flexural strength of the cement and geopolymer mortars is shown in Figure 4.47 and Figure 4.48, respectively. The mixes with MS outperformed the mixes with POCS in both mortar types, which could be attributed to the lower stiffness of POCS. Furthermore, POC concrete is reported to have a lower modulus of elasticity compared to conventional concrete (Huda et al., 2016), which reduces its bending capacity. Nevertheless, in another study by Mohammed et al. (Mohammed et al., 2014), a POCS concrete beam produced comparable flexural strength to that of lightweight concrete.

On the other hand, the geopolymer mixes produced a relatively higher flexural strength than those of cement mixes. This trend was also observed by other researchers (Nath & Sarker, 2017), thereby confirming the better performance of geopolymers in flexure. Another plausible argument could be the inclusion of GGBS in the mixes. A recent study (Nath & Sarker, 2017) showed that the presence of GGBS would have a positive effect on the flexural strength of the mixes.

The comparison of the flexural strength of different POCS grades in both mortars shows a reduction in the strength as the particles get finer with POCS 0.15 - 0.60 (G-5 and G-10) achieving the lowest strength of all. In comparison, the cement and geopolymer mortars incorporating POCS 0.15 - 4.75 (G-2 and G-7) acquired the highest flexural strengths of 8.3 and 9.5 MPa among the POCS mixes, respectively. It was reported that coarser sand particles produce a more deformable and ductile mortar (Haach et al., 2011), which has a direct effect on the flexural behaviour of the mortar.

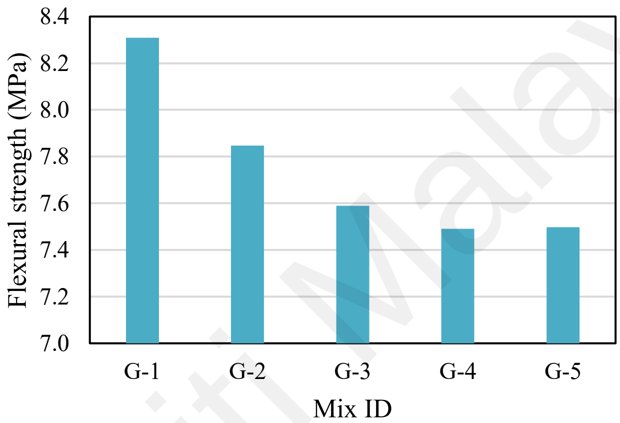

Figure 4.47: Flexural strength of cement mortar specimens

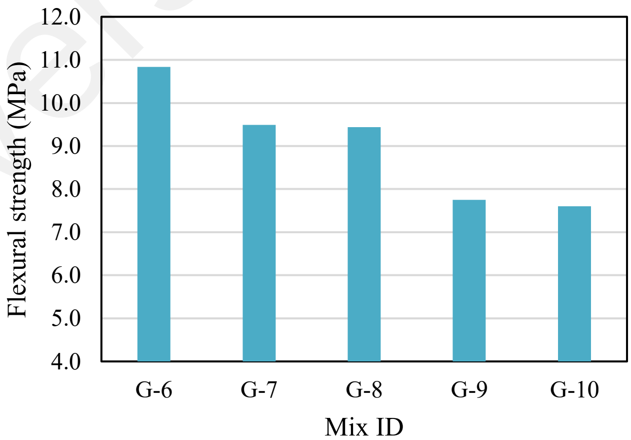

Figure 4.48: Flexural strength of geopolymer mortar specimens

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4.3.1.5 Drying shrinkage of POCS grading

The curves depicted in Figure 4.49 and Figure 4.50 represent, respectively, the drying shrinkage results of the cement and geopolymer mortars incorporating different POCS gradings. The majority of the shrinkage occurred in the first three months, followed by a gradual increase to the end of the period. The geopolymer specimens seem to be more affected by the shrinkage than the cement mortar. The higher values for drying shrinkage in the geopolymer samples compared to the cement samples in this study could be due to the presence and contribution of GGBS in the reaction process. GGBS generates more heat in the reaction (Qiang et al., 2016), and, thus, with the increased generated C-S-H gel, higher drying shrinkage could occur in the geopolymer specimens (Jiang et al., 2018). Lee et al. (2006) experienced the same upward trend with the increase in the GGBS levels.

The sand particle size has a substantial effect on the drying shrinkage of the mortar. It can be observed that lower drying shrinkage values have occurred in coarser POCS particles in both the cement and geopolymer mortars. The lowest shrinkage happened with the incorporation of POCS 0.15 - 4.75 (G-2 and G-7) compared to other POCS gradings. A similar result was obtained by other researchers (Rao, 2001), thereby confirming the positive impact of the inclusion of coarser fine aggregates in terms of drying shrinkage.

The comparison between the behaviour of MS and POCS in drying shrinkage shows that MS had a relatively lower shrinkage. However, in the geopolymer mixes, POCS 0.15 - 4.75 (G-7) mortar demonstrated better performance in terms of drying shrinkage with a value that was almost 40% lower than that for MS mortar.

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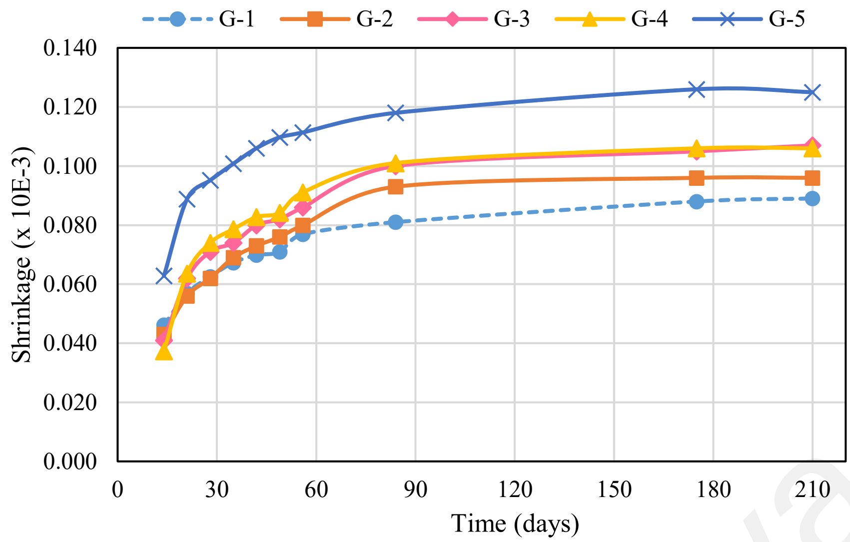

Figure 4.49: Drying shrinkage of cement mortar samples

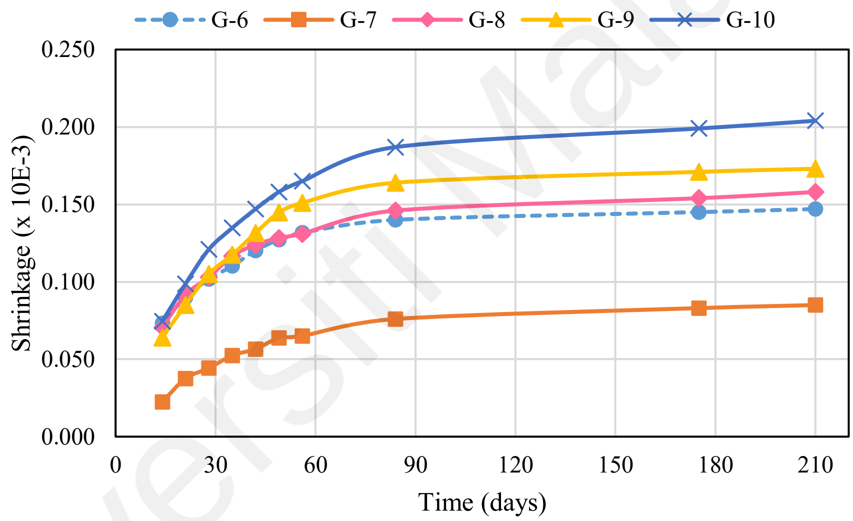

Figure 4.50: Drying shrinkage of geopolymer mortar samples

4.3.1.6 Water absorption and sorptivity of POCS grading

The properties of sand could substantially affect the water absorption and sorptivity (rate of water absorption) of the mortar since the majority of the mortar's volume is occupied by sand. The water absorption test is considered as a means of evaluating the durability of cementitious materials (Castro et al., 2011), and it indicates the pore level of the specimens; higher rates could demonstrate a higher level of porosity. The water absorption and sorptivity tests were conducted on the cube specimens after 28 days of curing. Water absorption results of cement and geopolymer mortars are shown in Figure

4.51 and Figure 4.52, respectively. MS mortar acquired the lowest water absorption rate (8%) among other cement mortars ranging from 10 to 11%. The porous POCS could be considered as the main reason for the higher water absorption level compared to that of MS mortar.

The water absorption value for geopolymer mortars was recorded at 8-10%, which is around 1-2% lower than that of the cement mortars. The lower water absorption of geopolymers could indicate the denser composition of the geopolymers compared to the conventional mortar. This result is in line with the density of the specimens (Figure 4.46), which showed higher densities of geopolymer mortars compared to the cement ones. Another reason for the lower values of the geopolymers could be the use of GGBS, which contributes to the reduction in the water absorption value (Mo et al., 2016).

The comparison between the performance of different POCS gradings in both mortar types reveals that smaller sand particles produce mortars with lower water absorption rates. This could mainly be attributed to the more compacted structure of the mortars with the incorporation of smaller sand particles. Overall, the performance of POCS geopolymer mortar in terms of water absorption was similar to that of MS, while the mixes with finer POCS demonstrated better performance.

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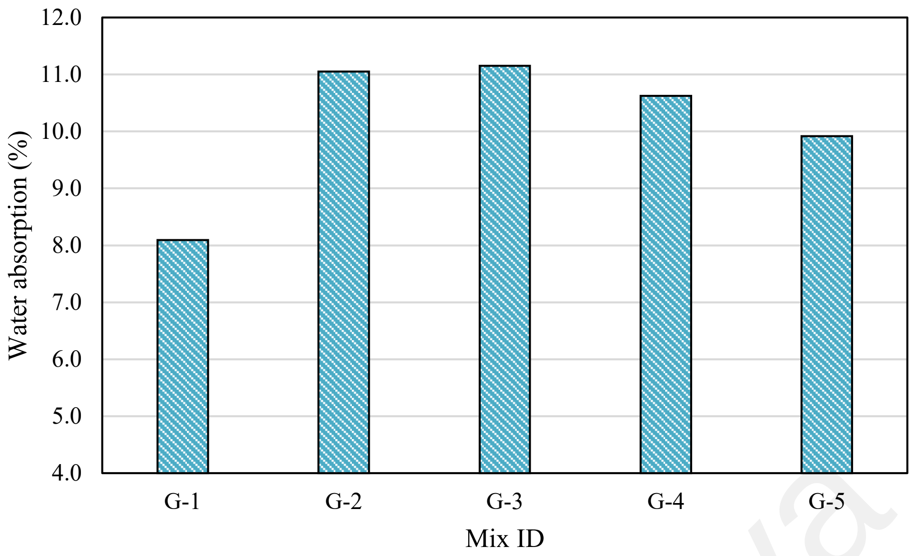

Figure 4.51: Water absorption of cement mortar

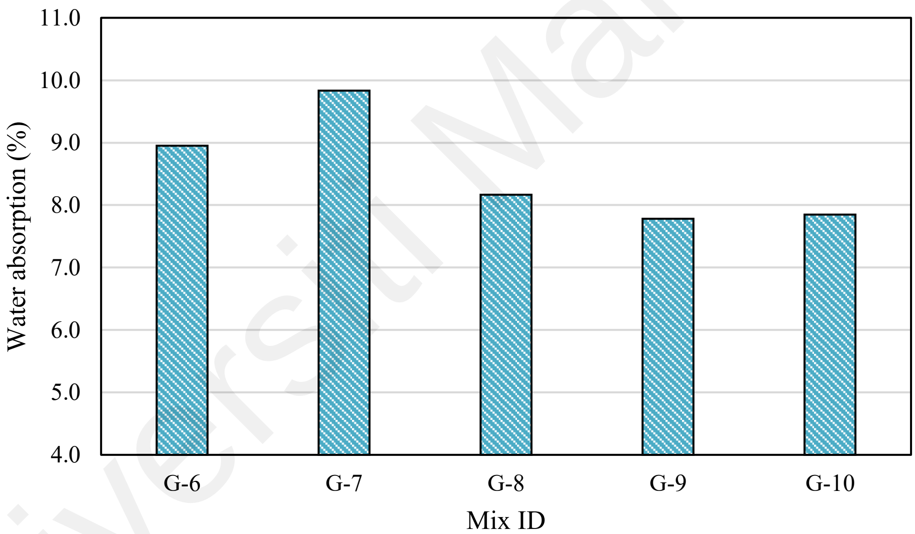

Figure 4.52: Water absorption of geopolymer mortar

The sorptivity of the mortars shown in Figure 4.53 and Figure 4.54 demonstrates that all the geopolymer mortars experienced lower values compared to the same grading of the cement mortars, which is in line with the results for the waster absorption. This could be ascribed to the fact that geopolymers inhibit the interconnectivity of the pore structures since the geopolymer gel contains a much denser matrix (Ng et al., 2018). The comparison between the trends in the cement mortars, shows that, unlike the POCS specimens, the MS mortar absorbed the majority of the water in the first four hours, while, for the POCS samples, the slope of the increase was lower but constant during the whole period. In both

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the geopolymer and cement mortars, POCS 0.15 - 2.36 (G-3 and G-8) absorbed the lowest amount of water in 24 hours compared to other POCS gradings with a comparable result to that of cement mortar utilizing MS.

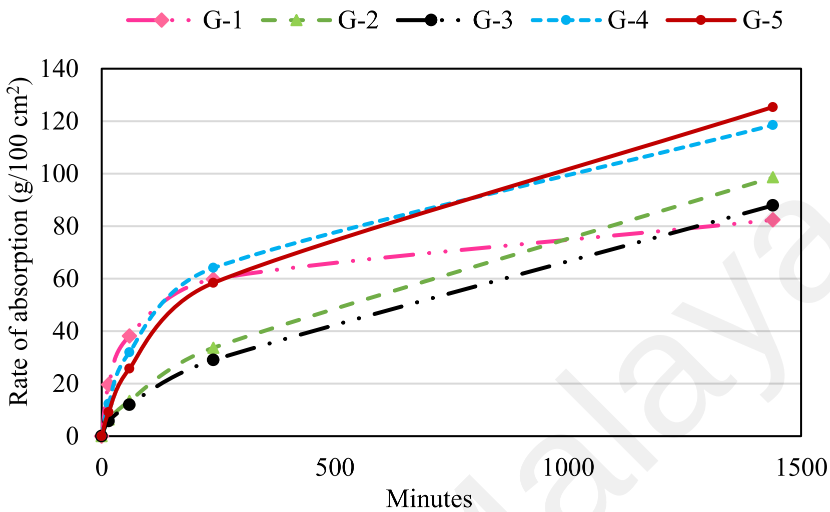

Figure 4.53: Sorptivity of cement mortar

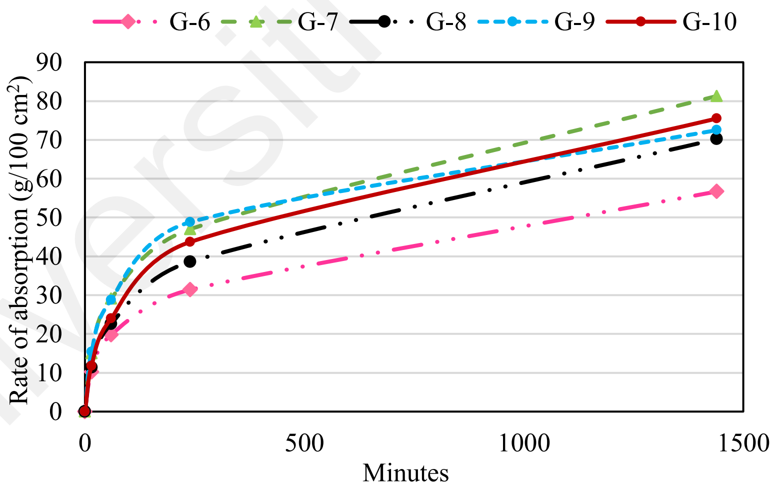

Figure 4.54: Sorptivity of geopolymer mortars

4.3.1.7 SEM analysis of POCS grading

SEM microstructural images were used to investigate the possible effect of the sand grading on the interconnection of sand particles with the paste on the geopolymer mixes utilizing POCS 0.15 - 4.75 (G-7) and POCS 0.15 - 0.60 (G-10). As shown in Figure 4.55, in the mix with POCS 0.15 - 4.75, there are unfilled layers that have not been filled. However, the SEM images demonstrate that the majority of the pores are filled in the

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POCS 0.15 - 0.60 geopolymer mortar. This could lead to the higher compaction and denser structure of the POCS 0.15 - 0.60 specimens reported in Figure 4.46, and it correlates well with the increased compressive strength with finer sand particles. However, as can be seen from images (c) and (d), microcracks have appeared on the samples, which could be one of the main reasons behind the increased drying shrinkage of the mortars incorporating the POCS 0.15 - 0.60. The increased drying shrinkage in the current research is in line with that of Venkatarama Reddy and Gupta (2008) with a similar cause.

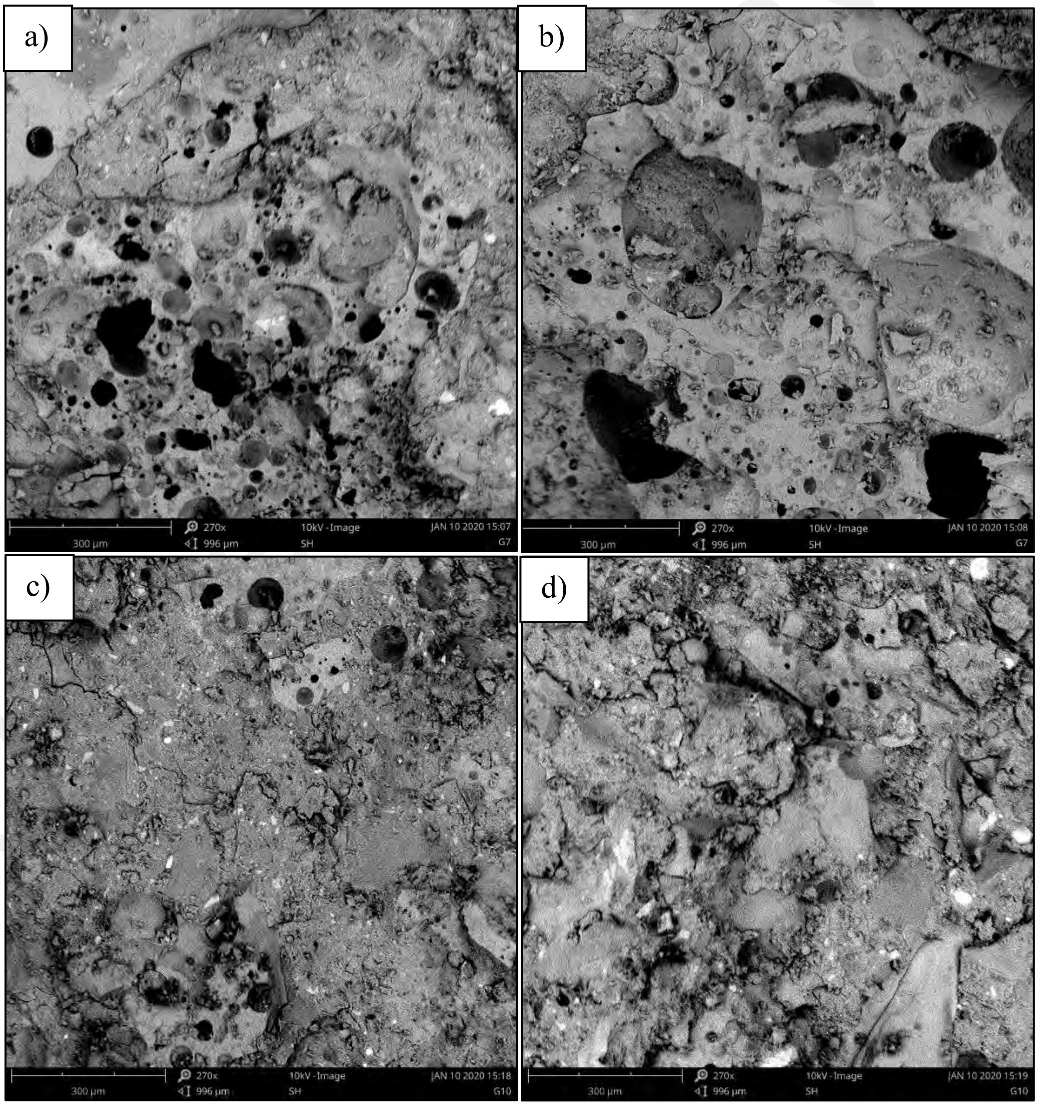

Figure 4.55: SEM images: a & b) G7, c & d) G10

CHAPTER 5: EFFECT OF BATIK WASTE INCORPORATION ON THE PROPERTIES OF GEOPOLYMER MORTAR AND APPLICATION

5.1 Geopolymer mortar with the incorporation of batik waste

In this study, the batik waste was used as the sodium silicate and free water replacement in geopolymer mortar. Commercial sodium silicate was used as the control material and was replaced with the batik waste with increments of 20% up to 100%. An equal blended mix of FA and GGBS was used as the binder as shown in Table 5.1. Six mixes were produced to evaluate the performance of batik waste in geopolymer in terms of mechanical properties, chemical resistivity, and microstructural analyses. All the mixes were cured in the ambient condition. The mix proportions used in this research are shown in Table 5.1.

Table 5.1: Mix proportions of geopolymer mortar using batik waste

		Proportions (kg/m^3)					
ID	Replacement (%)	Sand	FA	GGBS	NaOH	Silicate	Batik waste
B-1	0	1391	328.3	328.3	131.3	197.0	0
B-2	20	1391	328.3	328.3	131.3	157.6	39.4
B-3	40	1391	328.3	328.3	131.3	118.2	78.8
B-4	60	1391	328.3	328.3	131.3	78.8	118.2
B-5	80	1391	328.3	328.3	131.3	39.4	157.6
B-6	100	1391	328.3	328.3	131.3	0	197.0

The workability of the mixes was monitored through the flow table test to obtain a flow of 110 ± 5%. In order to achieve and keep the desired flow for all the mixes, additional water was added. However, the amount of additional water needed for increasing the flow to 110, was decreased with the increase in the silicate replacement with batik waste. This was due to the presence of water in the batik waste, which consequently provided sufficient workability without the need for excess water in the mix

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using 100% batik waste. Table 5.2 shows the amount of additional water required for the mixes, which is reported in percentage of the binder.

Table 5.2: Additional water for the mixes with batik waste

Mix ID	Batik waste (%)	Additional water (%)
B-1	0	12
B-2	20	10
B-3	40	7
B-4	60	5.5
B-5	80	4
B-6	100	0

5.1.1 Density of batik waste mixes

According to Figure 5.1, the 28-day oven-dry density of the mixes experienced a sharp decrease with the incorporation of the batik waste; the density of the control mix dropped from 2003 to 1933 kg/m^3 with 100% replacement of the sodium silicate. The governing factor in the reduction of the density could be the higher specific gravity of the commercial sodium silicate compared to the batik waste. Nevertheless, the influence of the additional free water should not be neglected as it has a reverse effect on the density.

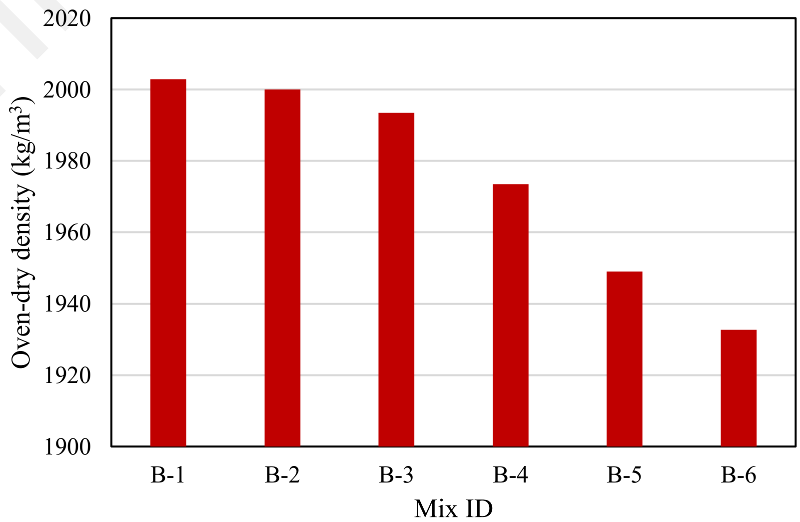

Figure 5.1: Density of geopolymer mixes using batik waste

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5.1.2 Compressive strength of batik waste mixes

The cube specimens were evaluated in terms of compressive strength at the ages of 1, 7, 28, and 90 days. According to the results shown in Figure 5.2, with the replacement of sodium silicate with the batik waste, the compressive strength experienced almost no significant change with up to 60% of replacement. The 28-day strength of B4 mix was 65.6 MPa while the control mix without any batik waste produced 68.8 MPa, which is less than 5% higher. Nevertheless, with the higher replacement of the silicate, B-5 and B-6 with the batik waste, the strength reduced drastically to reach 35.7 and 19.1 MPa in 28 days, respectively.

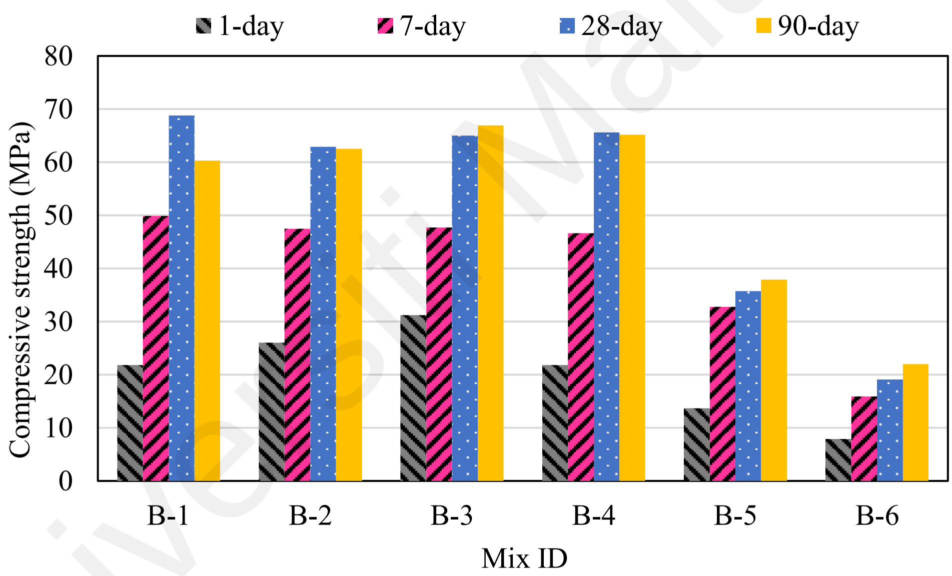

Figure 5.2: Compressive strength of geopolymer mixes using batik waste

The significant decrease in the strength with 80 and 100% replacement could be ascribed to a low amount of sodium silicate, which reduced the level of reactivity and alkalinity of the geopolymerization. Kamseu et al. (2017) found that with the increase in the alkalinity, the dissolution of the mixes could be improved, resulting in a denser product. Thus, the decrease in the strength and the density of the mixes could be caused by weakened polycondensation.

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It is evident from Figure 5.2 that the strength of the control mix (B-1) along with B-2, B-3, and B-4 was not developed properly between 28 and 90 days and they mostly experienced a reduction of strength in the period. A similar result was achieved in a study using FA-GGBS mixes in which the strength was reduced after a certain point in the mixes with higher content (Li & Zhang, 2019). However, B-5 and B-6 did not experience a reduction in the 90-day strength as the degree of the geopolymerization and thus the excess heat was reduced.

5.1.3 Water absorption of batik waste mixes

Water absorption test was conducted to verify the results achieved in the previous sections. As depicted in Figure 5.3, the water absorption of the geopolymer mixes using batik waste was recorded between 7.9 and 10.7%. It can be understood that B-4 with 8.4% of water absorption had a satisfactory performance with only a 0.2% increase compared to the control mix while B-5 and B-6 had an increase of 1.9 and 2.5%, respectively. This could verify that the incorporation of up to 60% of the batik waste in the geopolymer mortar could not impact the integrity and performance of the mixes.

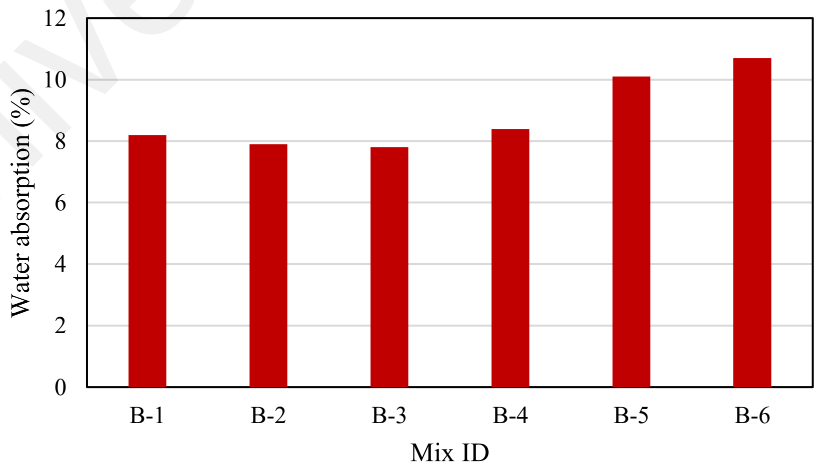

Figure 5.3: Water absorption of geopolymer mixes using batik waste

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5.1.4 XRD analysis of batik waste mixes

The diffraction pattern of the XRD analysis on the batik waste mixes is shown in Figure 5.6. Three mixes were chosen to be analysed using XRD: B-1 with 100% commercial sodium silicate, B-4 as the optimum mix with 60% of the batik waste, and B-6 using 100% replacement of the silicate and free water with the batik waste. The analysis was performed on the geopolymer paste at the age of 28 days.

According to the diffractions, all three mixes produced almost identical peaks. However, the periclase has emerged more dominantly in the B-4 mix at 38 and 44 degrees compared to the B-1 and B-6 mixes. Periclase (MgO) is a magnesium-based mineral that could preserve its stability even in the high pressures. The addition of reactive MgO in the geopolymer paste could improve the performance of the paste by reducing the shrinkage of the specimen (Li et al., 2019c).

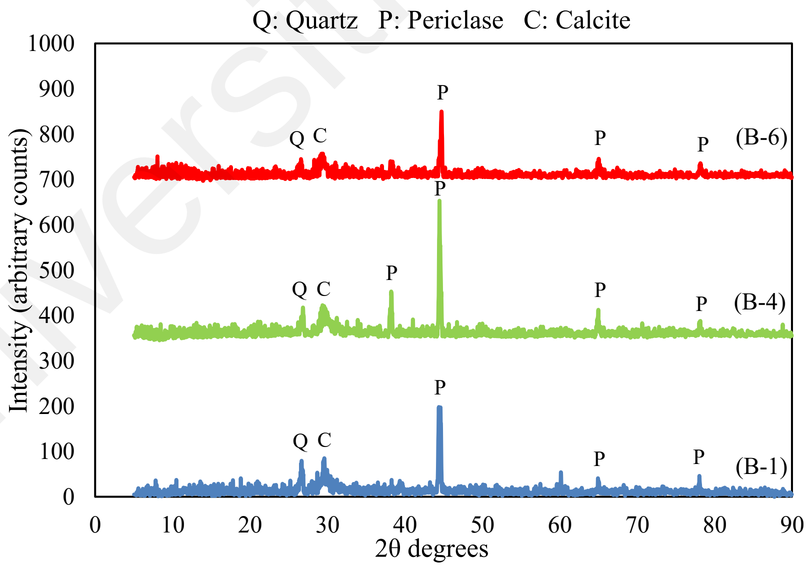

Figure 5.4: XRD analysis on the paste using batik waste

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5.1.5 Chemical resistance of batik waste mixes

The geopolymer mix using 60% of batik waste (B-4) was chosen as the optimum mix for evaluation against magnesium sulfate and HCl acid solutions for immersion of 90 days. The 28-day compressive strength of the mix was used as the initial strength (before immersion). Figure 5.5 demonstrates the residual strength of the cube specimens after immersion in the sulfate solution. Overall, the strength was reduced from 65.6 to 55.2 MPa over the 90-day period and thus, 16% decrease was experienced. Nevertheless, the reduction of the geopolymer mortar without using the batik waste was observed in Figure 4.37 as 17% during the same period. This signifies that the inclusion of the batik waste did not affect the resistance of the mix against sulfate solution.

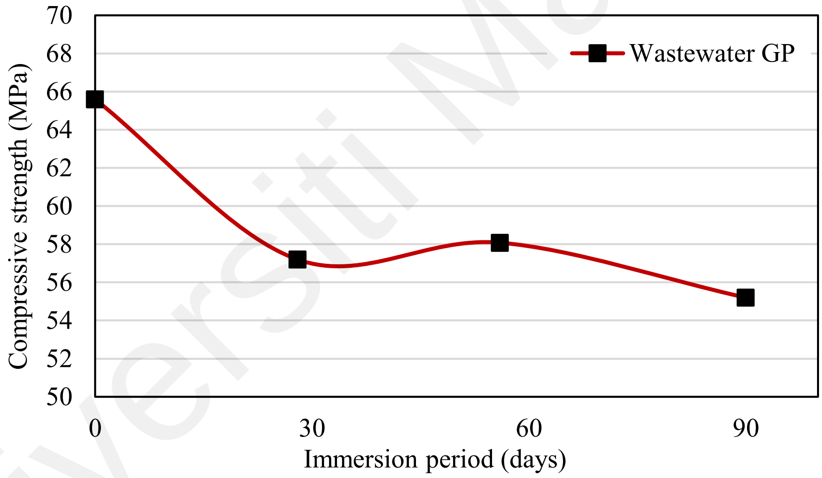

Figure 5.5: Sulfate resistance of geopolymer mortar using batik waste

Moreover, according to Table 5.3, the UPV values of the specimens immersed in the sulfate solution have been recorded as 3.84, 3.43, and 3.41 km/sec for periods of 28, 56, and 90 days, respectively, which fall in the good range even after the immersion.

The residual compressive strength of the geopolymer mortar cube specimens using the batik waste is shown in Figure 5.6. The influence of the acid solution was more significant on the specimens compared to the magnesium as 40% of the strength was reduced for the acid samples during the 90-day period with an almost constant steep. A similar study on

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the effect of HCl acid solution on the geopolymer mortar showed a 38% reduction in 90 days (Vafaei & Allahverdi, 2019).

UPV result (Table 5.3) of the acid specimens was recorded lower to that of sulfate specimens as well; the acid solution had a more deleterious effect on the specimens. However, in both cases of acid and sulfate solutions, the specimens produced UPV values of above 3.0 km/s after 90 days of immersion which is reported to be in the satisfactory range.

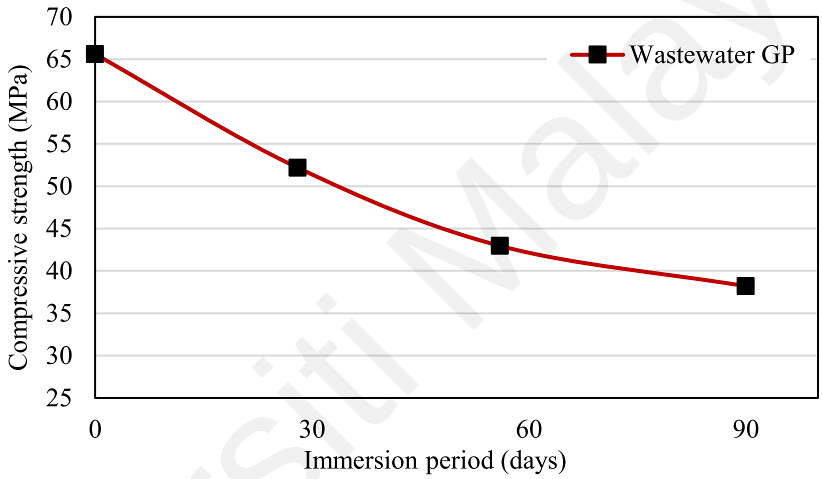

Figure 5.6: Acid resistance of geopolymer mortar using batik waste

Table 5.3: UPV values of chemical resistance specimens

	UPV (km/s)		
	28-day	56-day	90-day
Sulfate samples	3.84	3.43	3.41
Acid samples	3.25	3.11	3.01

5.2 Application

The findings and methodologies of the current study have been employed to develop geopolymer concrete and block prototypes to promote its potentiality and sustainability. For this purpose, with the incorporation of palm oil clinker as replacement of conventional fine and coarse aggregates as well as the incorporation of batik waste, a sustainable geopolymer concrete was developed and evaluated. Furthermore, by utilizing a dry mix, lightweight geopolymer blocks were developed using a hollow block machine.

5.2.1 Geopolymer concrete

Four mixes were used in this section, as described in Table 5.4.

Table 5.4: Concrete mix details

Mix ID	Concrete type	Fine agg.	Coarse agg.	Sodium silicate
C-1	Geopolymer	MS	Crushed granite	Commercial
C-2	Geopolymer	POC	POC	Commercial
C-3	Geopolymer	POC	POC	60% batik waste
C-4	Cement	MS	Crushed granite	N/A

The slump test was performed prior to the casting to ensure the mixes have enough workability of 60-180 mm (Table 5.5).

Table 5.5: Slump value of the concrete mixes

Mix ID	Slump (mm)
C-1	80
C-2	130
C-3	150
C-4	130

Cubes, cylinders, and prisms were prepared and cured in the ambient condition and in the water tank for the geopolymer and conventional concrete specimens, respectively. The compressive strength and UPV values of the specimens are shown in Figure 5.7 and Figure 5.8. According to the figures, the geopolymer concrete with the conventional aggregates produced the highest compressive strength with the 28-day value of 71 MPa followed by the 53 MPa for the cement concrete mix.

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Nevertheless, C-2 and C-3 with the incorporation of 100% replacements of fine and coarse aggregates by POC, produced 28-day strengths of 26 and 32 MPa which have surpassed the minimum required strength for structural purposes (17.2 MPa) introduced by American concrete institution (Committee, 1995).

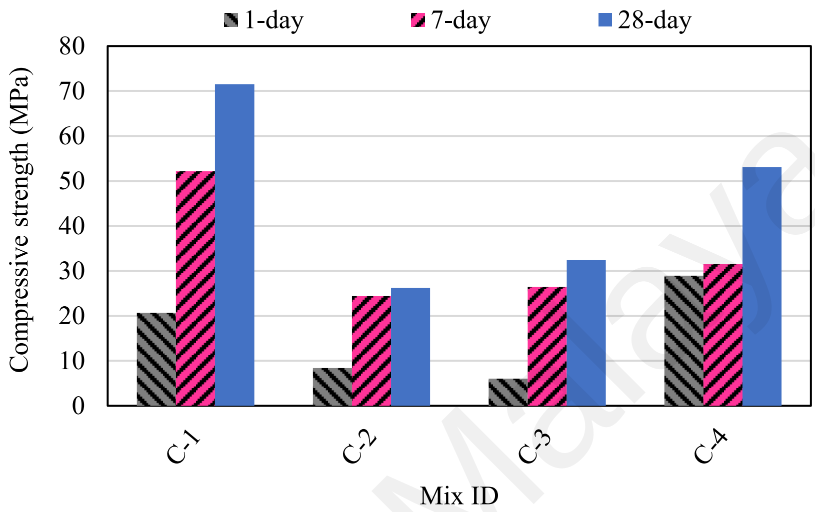

Figure 5.7: Compressive strength of concrete specimens

Interestingly, the usage of batik waste in the concrete has improved the performance of the mix in different aspects. The strength and UPV values have increased by 24 and 6%, respectively. This could be correlated to the increased lubrication between the layers of the POC particles in the mix using the batik waste. With the increased flowability, the paste could penetrate the POC particles more efficiently and increase the strength of the bond and contribute to the reduction of the porosities.

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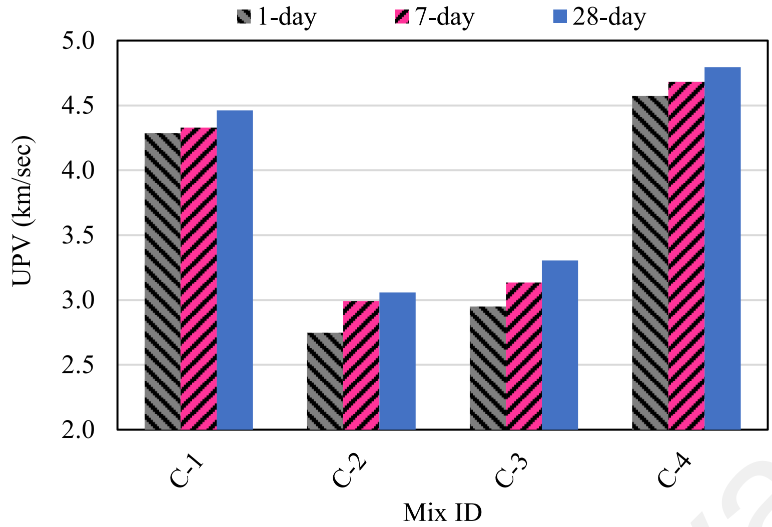

Figure 5.8: UPV result of concrete specimens

Figure 5.9 and Figure 5.10 demonstrate the 28-day splitting tensile and flexural strength of the specimens. The trend of both graphs resembles that of the previous section; C-1 and C-4 produced higher values compared to the other two mixes. The cement and geopolymer mixes experienced the same result in the splitting tensile test while the cement concrete produced slightly higher flexural strength. The results of previous studies on utilizing POC fine and coarse aggregates in conventional concrete were in line with the findings of this study and showed a reduction in the splitting tensile strength with an increase in the POC level (Abutaha et al., 2018; Ahmmad et al., 2017).

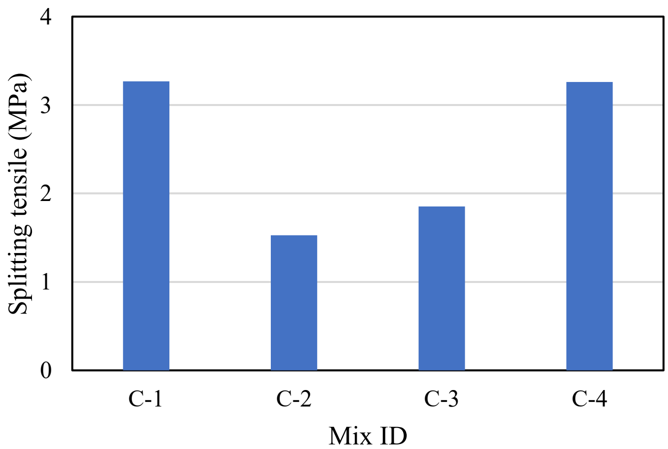

Figure 5.9: Splitting tensile of concrete specimens

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Incorporation of 60% batik waste improved the tensile and flexural strengths by 27% and 20%, respectively. The flexural strength achieved by C-1 and C-4 were 6.4 and 7.3 MPa, respectively, which could be considered as high-performance concrete (Wille et al., 2011) since the samples were not reinforced with fibres. On the other hand, the flexural strength produced by C-2 and C-3 can be categorized as average flexural strength (3-5 MPa).

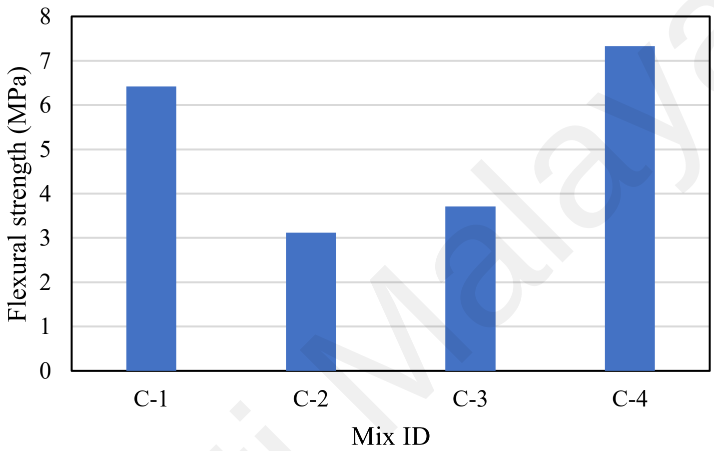

Figure 5.10: Flexural strength of concrete specimens

The electrical resistivity of the concrete was measured using the Resipod equipment which operates based on the principle of the Wenner probe. Cylinder specimens were used for this purpose after 28 days of curing. The electrical resistivity could be an indication of the corrosion rate of the concrete and its possible resistance to aggressive environments. According to the results shown in Figure 5.11, the conventional concrete demonstrated the best performance among all the mixes with 12.8 kΩ cm. Based on the resistivity measurement of Wenner 4-probe system, the electrical resistivity of 10-20 kΩ cm shows that the concrete has a corrosion rate of low to moderate while with a resistivity of 5-10 kΩ cm, the corrosion rate is higher.

One significant reason for the lower electrical value of the geopolymer specimens is because of its different curing method to that of conventional concrete. Since the reading

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is based on the ions in the pore liquid, and the geopolymer concrete specimens were not immersed in the water during the curing time, lower reading was obtained. Moreover, the porosities present in the POC specimens could not be denied as it directly affects the electrical resistivity of the samples. However, the resistivity was increased with the incorporation of the batik waste.

According to the previous studies (Hope et al., 1985), resistivity values of higher than 8.5 kΩ cm could be less exposed and affected by corrosion. Thus, mix C-3 has demonstrated satisfactory performance in terms of corrosion-resistance.

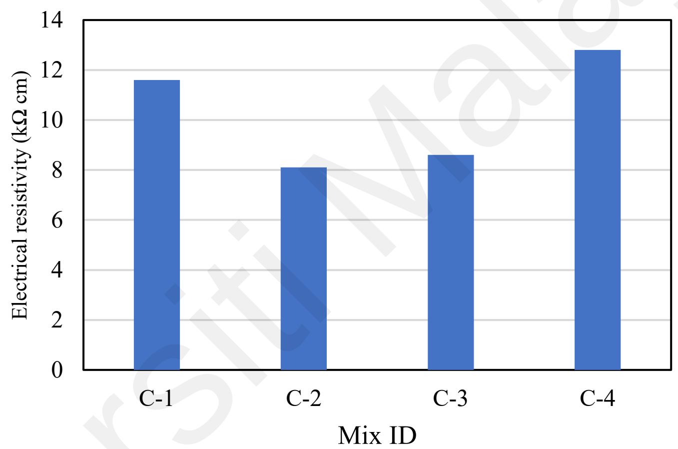

Figure 5.11: Electrical resistivity of concrete specimens

The density of the concrete cube specimens was measured, and it is shown in Figure 5.12. As can be observed from the graph, there is a massive difference in the density of the concrete mixes using conventional aggregates (C1 and C4) and the concrete mixes using POC aggregates (C2 and C3). The incorporation of POC fine and coarse aggregates caused a reduction of about 30% of the density of the concrete.

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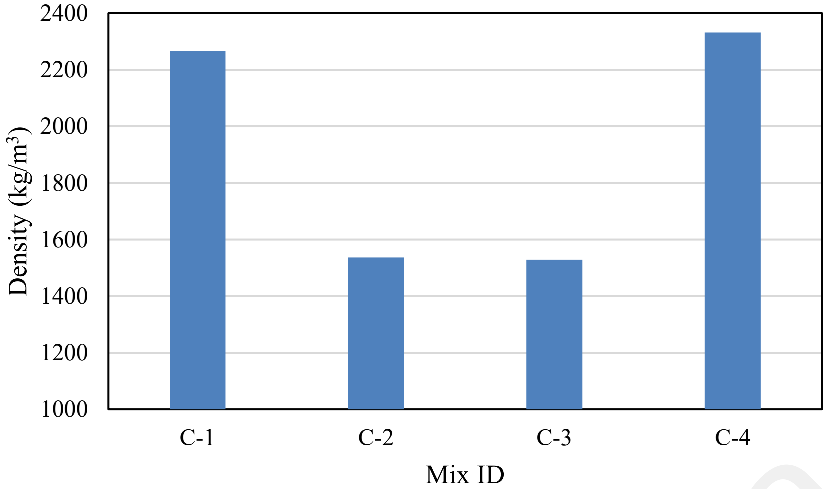

Figure 5.12: Oven-dry density of concrete specimens

As expected, the water absorption test showed higher values for the POC concrete specimens, which is similar to the results achieved for the POC mortar. Figure 5.13 shows the water absorption values for the concrete specimens in which the conventional concrete had the lowest absorption rate of 2.6%, followed by the geopolymer concrete using conventional aggregates with 3.6% of water absorption. Kanadasan et al. (2018) concluded that the waster absorption value of the mix containing 100% POC as the fine aggregate was 39% higher than the mix without palm oil clinker as the sand.

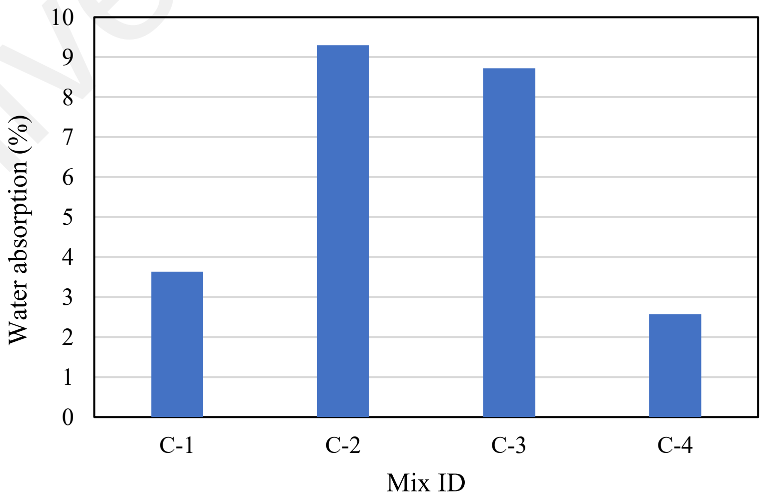

Figure 5.13: Water absorption of concrete specimens

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5.2.2 Geopolymer hollow block

With the utilization of 100% palm oil clinker as fine and coarse aggregates, a geopolymer block was designed and evaluated. A mixture of FA and GGBS was used as the binder with 85 and 15% of incorporation, respectively. The blocks were cured in the ambient condition and shown in Figure 5.14.

Figure 5.14: Geopolymer hollow block

The compressive strength of the blocks was determined at the age 7 and 28 days, and it is summarized in Table 5.6 among with the 28-day density using the net area of the blocks.

Table 5.6: Geopolymer hollow block results

	OD density (kg/m^3)	7-day strength (MPa)	28-day strength (MPa)
Geopolymer block	1597.6	6.4	7.1

As per guidelines for non-loadbearing concrete masonry hollow blocks (ASTM International, 2014c), the minimum required compressive strength is 4.14 MPa, and the density classification of the oven-dry hollow blocks is depicted in Table 5.7. Since the geopolymer hollow block using POC fine and coarse aggregates has produced a 28-day strength of well above the minimum requirement of ASTM and the oven-dry density of

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the block was found 1597.6, it could be categorised as a standard lightweight non-loadbearing hollow block.

Table 5.7: Density classification of non-loadbearing blocks (ASTM International, 2014c)

Density classification	OD density (kg/m^3)
Lightweight	Less than 1680
Medium Weight	1680 – 2000
Normal Weight	More than 2000

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CHAPTER 6: CONCLUSIONS AND FURTHER RECOMMENDATIONS

This chapter includes a summary of the overall findings and recommendations for further studies based on the outcome of the current research. The majority of the literature focused on the replacement of the palm oil clinker wastes using the weight-based calculation in which a high volume of the binder was needed to produce a comparable strength to that of control mix. Moreover, the replacement of conventional fine and coarse aggregates with palm oil clinkers in geopolymer mortar/concrete produced high-performance specimens in terms of mechanical properties and durability aspects.

Although the control specimens using conventional materials demonstrated better performances in some categories, the importance of the usage of waste materials in practical and feasible applications should not be neglected as it increases the sustainability of the concrete, one of the world's most used substances on the planet. Even the tiniest improvements in terms of preservation of the Earth's virgin materials could have an influential impact. Furthermore, with the increased knowledge on the geopolymers and its numerous advantages, it could enable the widespread production of a sustainable concrete without the incorporation of cement, known as a massive CO_2 emitter. The widespread production of geopolymers could contribute to the reduction of the overall prices as well.

6.1 Conclusions

In this chapter, a summary of the conclusions and recommendations for future studies have been presented.

6.1.1 Incorporation of POCS in cement mortar

The following conclusions were derived from the cement mortar incorporating POCS:

- The rough and poriferous structure of POC contributes to the enhancement of a better bond with the cement paste.
- The use of POCS as conventional sand replacement reduces the SSD density of the final mortar between 12 and 15%.
- Although the volume of paste was maintained for both the POCS and control-MS mortar, the comparison of these two mortars shows a reduction in the 28-day compressive strength of about 13% for the former.
- Ultrasonic pulse velocity results showed well-compacted specimens for POCS mortars, as the velocity of the POCS mortar was recorded at 4.09 km/sec; nevertheless, this is slightly lower compared to the control sample.
- Increasing the paste content in the POCS mortar increases the strength to a certain extent. Increasing the paste content to above 47% by mass resulted in a reduction in the strength and UPV.
- The maximum strengths achieved for w/c ratios of 0.50, 0.55, and 0.60, were recorded with the incorporation of 47, 47, and 45% of the paste mass, respectively.
- The microscopic view of the crushed cubes revealed that failure occurs at both POCS and the bond between the paste and POCS due to the high stiffness of the POCS.
- The cost comparison between the control mix and the mix using POCS showed that the replacement of MS with POCS could reduce the total cost of the mortar by 16%. Moreover, M3 mix proposed in this study provided a better (bi) index compared to the previous studies using POCS. Although the (bi) index of the MS mortar was lower than all those of POCS mortar, the incorporation of POCS in mortar would pave the way for a meaningful reduction in the

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depletion of natural resources and also deal with disposal issue of waste materials.

6.1.2 Incorporation of POCS in geopolymer mortar

- Increasing the molarity of the sodium hydroxide and GGBS content reduced the flowability of the geopolymer mortar.
- Study on the effect of NaOH molarity showed that with the increase in molarity from 8 to 14, the overall performance of the POCS geopolymer mortar, namely compressive strength, UPV, water absorption, and the sorptivity was improved. Conversely, 16-molar NaOH reduced the properties.
- Replacement of the MS with POCS in geopolymer mortar reduced the density of the cubes by about 17% while the replacement of FA with GGBS in the mixes had a slight increase in the density.
- The highest 28-day strength obtained for the oven-cured specimens was 50 MPa with 30% of GGBS, while for the ambient-cured specimens, it was 53 MPa with 50% of GGBS. Incorporation of GGBS in the mixes contributed to the strength development, UPV, reduction of water absorption, and sorptivity. Further, it reduced the dependency on the oven-curing method. The early strength development of mixes with GGBS within the first 7-days was about 62-85% of the 28-day strength for the ambient-cured specimens.
- The setting time of the 50% GGBS geopolymer mortar was found about 30 minutes for ambient-cured mixtures, which subsequently increased to an hour when GGBS was reduced to 30%.
- Geopolymer mortars showed higher resistance to the HCl and magnesium sulfate compared to cement mortar as the latter showed deterioration since the C-S-H gel present in the cement matrix is vulnerable to chemical attacks. The residual compressive strengths of POCS geopolymer mortar after 180 days of

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immersion in HCl and magnesium sulfate were found as 36 and 70% of the 28-day strength, respectively.

- The X-Ray diffraction and EDX analyses showed enhanced formations of Si-O-Si and C-A-S-H bonds in GGBS incorporated mixes, and the presence of calcite mostly in the ambient-cured mixes resulted in better mechanical properties.

6.1.3 Effect of grading of POCS in cement and geopolymer mortars

- The flow of the fresh mortar decreased through increasing the fineness of the POCS particles, and the reduction was almost proportionate for both the cement and the geopolymer.
- The compressive strength of POCS mortars increased with the incorporation of finer sand particles ascribing to the better compaction of the specimens, which reached the highest strength of 41 MPa for the cement and 63 MPa for the geopolymer mortar with POCS 0.15 - 0.60 at the age of 180 days. The strength development had an upward trend for cement mortars over the whole period, while for the geopolymer mortars, the strength was decreased slightly from 90 to 180 days.
- The density of the POCS cement and geopolymer mortars was reduced by up to 20% in comparison with the MS mixes. Furthermore, the density of the mortars increased steadily with the incorporation of finer POCS.
- The highest flexural strength of POCS cement mortars was recorded 7.8 MPa for the POCS 0.15 - 4.75, with a 6% reduction compared to that of MS. For the POCS geopolymers, however, the overall trend was higher than cement mortars, with the highest being 9.5 MPa for POCS 0.15 - 4.75, which is 12% lower than the control mix using MS. The flexural strength of both mortar types decreased with the decrease in the sand particle sizes.

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- The mixes with coarser POCS particles outperformed the finer mixes in terms of long-term drying shrinkage. In geopolymers, higher values were observed than that of cement ones, while POCS 0.15 - 4.75 geopolymer mortar had a lower drying shrinkage compared to the mix with MS.
- The water absorption was lowered with the incorporation of finer POCS due to the denser structure to reach 9.9% and 7.8% for POCS 0.15 - 0.60 cement and geopolymer mortars, respectively. However, the sorptivity of the mix with POCS 0.15 - 2.36 was lower compared to other POCS mixes.
- Based on SEM images, the incorporation of finer POCS particles contributed to filling the micro-pore layers.

6.1.4 Batik waste incorporation in geopolymer mortar

- The incorporation of the batik waste in the geopolymer mixes reduces the required amount of water needed for producing a flow of 110%. With 100% incorporation of the batik waste, the sodium silicate and free water could totally be removed from the mix and produce a 28-day compressive strength of 19 MPa with a 3.5% reduction of the density compared to the control mix.
- With 60% replacement of the sodium silicate with the batik waste, comparable results were achieved to that of the control mix using the commercial sodium silicate.
- The residual strengths of the geopolymer mortar mix incorporating 60% of batik waste after 90 days of immersion in HCl acid and magnesium sulfate were found to be 38.2 and 55.2 MPa, respectively.

Palm oil clinkers could be used as the total replacement of conventional fine and coarse aggregates in geopolymer concrete and yet produce desirable engineering properties.

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Similarly, they could be incorporated in the production of lightweight non-loadbearing hollow blocks.

6.2 Recommendations for future studies

Following research ideas could provide a foundation for future investigations:

- The reactivity and incorporation of the palm oil clinker in the cement hydration reaction or geopolymerization could be further studied. This could help to understand the behaviour of the clinkers in the mixes more efficiently.
- The batik waste has excellent potential to be used in the geopolymer concrete, and there are still several factors such as separation of the sodium silicate from the batik waste that could be studied to optimise the utilisation of the batik waste in the geopolymers. With a more in-depth analysis of the chemical process of the batik waste in the geopolymers, the outcome could be significant in the future.
- In the current study, geopolymer concrete was developed using palm oil clinker fine and coarse aggregates as an application to the findings. However, with further investigation and a better understanding of the palm oil clinker coarse aggregates, the performance of the concrete could be enhanced.

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