# UTILISATION OF ALUM SLUDGE ASH IN MORTAR PRODUCTION

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# UTILISATION OF ALUM SLUDGE ASH IN MORTAR PRODUCTION

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

Faculty of Engineering and Green Technology Universiti Tunku Abdul Rahman

September 2019

# DECLARATION

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

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# APPROVAL FOR SUBMISSION

I certify that this project report entitled **"UTILISATION OF ALUM SLUDGE ASH IN MORTAR PRODUCTION"** was prepared by **NG YEE LENG** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Engineering (Honours.) Environmental Engineering at Universiti Tunku Abdul Rahman.

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# UTILISATION OF ALUM SLUDGE ASH IN MORTAR PRODUCTION

#### ABSTRACT

Alum sludge (AS) is a by-product of water treatment plants that uses aluminium salts as a primary coagulant. It is the most widely generated water treatment sludge worldwide. The disposal of alum sludge into landfills has became an environmental issue due to the enormous quantities generated and the associated costs of disposal to landfill. Meanwhile, the production of cement mortar is very energy and resources intensive. Therefore, this study aims to incorporate alum sludge ash (ASA) as a substitute to cement in mortar production, as it is a negative cost waste while contributing to sustainable development of building materials. In addition to ASA, ground granulated blast furnace slag (GGBFS) was also used as an additional binder to enhance the strength of the mortar. The study includes the investigation the effect of ASA incorporation on the physical, chemical, mechanical and durability properties of mortar. In this study, ten types of composite mortars are prepared, namely M-CTR, M-2ASA, M-4ASA, M-6ASA, M-2GGBFS, M-4GGBFS, M-6GGBFS, M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS. All the tests are conducted based on the BS EN 196-3, BS EN 12390-3, BS EN 1015 and BS 1881-122 standards. Life Cycle Assessment (LCA) was also conducted to check on the feasibility of using alum sludge ash to partially replace cement in mortar production. The laboratory results showed that the incorporation of ASA as a substitute to cement in the mortar production deteriorated its mechanical properties but its durability properties are improved.

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# LIST OF SYMBOLS / ABBREVIATIONS

$\mathbf{f}_{\mathbf{c}}$	Compressive strength, MPa		
Ι	Rate of water adsorption, %		
Pr	Porosity, %		
R	Flexural strength, MPa		
W.A.	Water absorption of hardened mortar specimen, %		
Al(OH) <sub>3</sub>	Aluminium Hydroxide		
$Al_2(SO_4)_3$	Alum		
$Al_2O_3$	Aluminium Trioxide/ Aluminium Oxide		
C=O	Carbonyl Group		
$C_2S$	Dicalcium Silicate		
C <sub>3</sub> A	Tricalcium Aluminates		
$C_3S$	Tricalcium Silicates		
$C_4AF$	Tertacalcium Aluminiferrite		
Ca(OH) <sub>2</sub>	Calcium Hydroxide		
Ca <sup>2+</sup>	Calcium ion		
Ca <sub>2</sub> SiO <sub>4</sub>	Dicalcium Silicate		
Ca <sub>3</sub> SiO <sub>5</sub>	Tricalcium Silicate		
CaCO <sub>3</sub>	Calcium Carbonate		
CaO	Calcium Oxide		
CaO•2SiO <sub>2</sub> •4H <sub>2</sub> O	Calcium Silicate Hydrate		
CaSO4	Gypsum		
С-Н	Hydrocarbon		
$CO_2$	Carbon Dioxide		
C-S-H	Calcium Silicate Hydrate		
Fe <sub>2</sub> O <sub>3</sub>	Iron (III) Oxide/Ferric Oxide		

0	e			
MgO	Magnesium Oxide			
Na <sub>2</sub> O	Sodium Oxide			
OH-	Hydroxide ion			
S=O	Sulphur Monoxide			
Si-O	Organosilicon			
SiO <sub>2</sub>	Silicon Dioxide			
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared			
	Spectrometry			
BS	British Standards			
CCS	Carbon Capture and Storage			
CF	Clinker Factor			
DOE	Department of Environment			
EC	Embodied Carbon			
EE	Embodied Energy			
EPA	Environmental Protection Agency			
GHG	Green House Gases			
IEA	International Energy Agency			
LCA	Life Cycle Assessment			
TOC	Total Organic Carbon			
AS	Alum Sludge			
ASA	Alum Sludge Ash			
CLMS	Controlled Low Strength Material			
GGBFS	Ground Granulated Blast Furnace Slag			
GGBS	Ground Granulated Blast Slag			
M-2ASA	OPC with 2 % ASA replacement as part of cement			
M-2ASA 4GGBFS	OPC with 2 % ASA and 4% GGBFS replacement as part of			
	cement			
M-2GGBFS	OPC with 2 % GGBFS replacement as part of cement			
M-4ASA	OPC with 4 % ASA replacement as part of cement			

Potassium Oxide

Magnesium Carbonate

K<sub>2</sub>O

MgCO<sub>3</sub>

M-4ASA 4GGBFS	OPC with 4 % ASA and 4 % GGBFS replacement as part of		
	cement		
M-4GGBFS	OPC with 4 % GGBFS replacement as part of cement		
M-6ASA	OPC with 6 % ASA replacement as part of cement		
M-6ASA 4GGBFS	OPC with 6 % ASA and 4% GGBFS replacement as part of		
	cement		
M-6GGBFS	OPC with 6 % GGBFS replacement as part of cement		
M-ASA GGBFS	Alum Sludge Ash Cement Mortar		
M-CTR	Control Mix		
OPC	Ordinary Portland Cement		
POFA	Palm Oil Fuel Ash		
SCM	Supplementary Cementitious Materials		
SF	Silica Fume		

# **CHAPTER 1**

#### INTRODUCTION

# **1.1 Introduction to Alum Sludge**

This report shows the study of the possibility of using alum sludge (AS) as a partial replacement of cement in producing mortar. The main purpose of this study is to investigate the development of engineering properties after up to 6 % of AS has been used as cement replacement. Alum is generally used as a primary coagulant in the production of clean drinking water where AS is a by-product of the processing of drinking water in water treatment plants (Yang et al., 2006). The management of AS has became an environmental issue due to the massive volume generated and the costs of disposal to landfill (Victoria A N, 2013). In Malaysia, AS is classified as scheduled waste, it cannot be discharged into streams and is governed by the Department of Environment (DOE, 2005). The Semanggar water treatment plant in Kota Tinggi, Johor alone has a 40 acre landfill area, accommodates approximately 120 tonnes of raw sludge per year (Paramalinggam et al., 2015). Hence, high volume of AS is being generated and dumped in the landfill.

# **1.2** Cement Production

The cement industry contributes to 5-7 % of global  $CO_2$  emissions. (Mintus et al., 2006). Cement production is a high raw resources and high energy intensive process consisting of raw material preparation (Stage 1), clinker production (Stage 2) and

grinding and blending (Stage 3) as illustrated in Figure 1.1. Cement manufacturing has always been ranked in the list of the main sources of carbon emissions among industrial activities. During clinker production, clinker is heated up to 1450 °C that accounts for the 60 % of carbon dioxide emission (Benhelal et al., 2012). Carbon dioxide is mainly released from combustion of huge amount of fossil fuels and decomposition of calcium carbonate to calcium oxide and magnesium carbonate to magnesium oxide as two main clinker components (Benhelal et al., 2012).

$$CaCO_3 \longrightarrow CaO + CO_2$$
 (1)



$$MgCO_3 \longrightarrow MgO + CO_2$$
 (2)

**Figure 1.1: Block Diagram of Cement Production Process** 

Utilising alternative materials is among the strategies of CO<sub>2</sub> reduction besides energy saving and carbon separation and storage (Benhelal et al., 2013). Energy saving approaches such as shifting from wet process to dry process reduces up to 50 % of required energy and mitigates almost 20 % of CO<sub>2</sub> emissions in the calcination process (Benhelal et al., 2013). Carbon capture and storage (CCS) is also an effective way to decrease release of CO<sub>2</sub>. However, due to technical, financial and regulation constrain, implementation of CCS is not foreseen before 2020 (Benhelal et al., 2013). To date, utilising industrial by-products make the greatest contribution in emissions mitigation in cement plants and landfills.

# **1.3** Composite Cement

Composite cement is a cement consisting of clinker, gypsum, pulverised fuel ash, blast furnace flag and limestone designated by the specifications of BS EN 197: 2000. Reuse of waste in cement production mainly depends on the chemical composition of the waste. AS is composed of high content of aluminium oxide Al<sub>2</sub>O<sub>3</sub> and silicon dioxide SiO<sub>2</sub> which is similar to conventional clay used in construction materials. Thermal treatment of alum sludge ash (ASA) produces tricalcium aluminates C<sub>3</sub>A and tricalcium silicates C<sub>3</sub>S compounds which are also commonly present in ordinary portland cement (Tantawy, 2015). The calcination products of AS which possess similar properties like cement could be applicable in the production of composite cement (Frías et al., 2014). Although calcination process improved the microstructure and enhanced the pozzolanic activity of AS (Didamony et al., 2014), but calcination associated with substantial energy consumption and carbon footprint.

#### **1.4 Waste Management**

Management and disposal of AS to landfill are global issue. Unlike sewage sludge, low calorific value of AS makes it impossible to recover energy from incineration treatment (Wang et al., 2018). At present, all AS are subjected to energy-intensive dewatering process followed by non-sustainable landfill disposal According to the Environmental Protection Agency (EPA), landfills alone have contributed to a total of 31.7 % in the greenhouse gas (GHG) emissions (EPA, 2016).

One of the approaches toward emission reduction in cement plant is by decreasing the ratio of clinker in cement named as clinker factor (CF) and substitute with suitable materials. While in 2003, the world average CF was 0.85, with South America as the lowest at 0.75 and North America as the highest proportion (Harder, 2006) at 0.92. Fly ash (by-product of fossil fuel power plants) and blast furnace slag (by-product produced in iron and steel production) are examples of by-product wastes that have been successfully commercialised to replace portion of clinker in cement production. By utilising industrial product as a portion of clinker in cement production,

it reduces the use of limestone and consequently mitigates CO<sub>2</sub> emissions resulted from limestone decomposition (Emad Benhelal et al., 2013).



Figure 1.2: Carbon Dioxide Reduction by utilising By-products a Supplementary Cementitious Materials in Cement Plant

Currently, there are no life cycle assessment (LCA) data regarding the use of ASA as partial replacement of cement in mortar production. Three important metrics are chosen to compare ASA with ordinary portland cement (OPC) on a sustainability basis. This includes the energy use, CO<sub>2</sub> emissions and cost. The LCA is performed to study the viable of partial replacement of alum sludge ash in cement to provide economical and environmental benefits. Therefore, AS has been chosen in research as a new cement material to tackle problems of disposal and convert it into an ecobeneficial building material for sustainable development.

# 1.5 Problem Statement

According to the report by Verlicchi,  $(2012) \ 1 \ m^3$ /s drinking water treatment plant generates about 8300 kg/day of sludge which means that the material is available in abundance. Exponential growth of human population increases the demand of clean drinking water. Aluminium salt are the most widely used coagulants for coagulation and flocculation process in water treatment plant. As a result, mass volume of AS generated in water purification process.

Wang et al. (2015) studied the surface chemistry and microstructure characteristics of sludge-derived Controlled Low Strength Material (CLSM) via microscope. They reported that high content of organic matter in AS would significantly delay the hydration of cement, which result in long setting time and low compressive strength (Wang et al., 2015). A previous work by Haider showed that calcination of AS at 800 °C for 2 hours in laboratory furnace would transform this inert kaolinite-based sludge into a metakaolinite based pozzolan (Haider et al., 2018). However, the usage of 800 °C to activate the sludge still consumed a lot of energy. Therefore, this present study focuses on characterisation of AS at 100 °C, its physical and chemical characteristics and their effects on the properties of the composite cement.

# 1.6 Objectives of Study

The objectives of this study are:

- 1. To assess the feasibility of using alum sludge as partial replacement of cement in mortar production.
- To study the physical, chemical, mechanical and durability properties of AS in mortar.
- 3. To evaluate the effectiveness of granulated ground blast furnace slag in achieving short stiffening time and suitable compressive strength.
- 4. To study the life cycle assessment (LCA) of alum sludge (AS) in mortar production.

# 1.7 Scopes of Study

The present research is designed to determine the feasibility of using ASA as partial replacement of cement in mortar production. The main materials used were ASA, ground granulated blast furnace slag (GGBFS), cement, water and sand. The engineering properties of mortar incorporated with ASA in terms of physical, chemical, mechanical and durability properties were studied. Overall, this study aims to produce a mix proportion for ASA cement based mortar as well as a trial mix mortar that is used as a control subjected to targeted strength of 12 MPa. Four types of mortar were prepared, namely: i) blank as control mix (M-CTR), ii) OPC with 2 %, 4 % and 6 % ASA replacement as part of cement (M-2ASA, M-4ASA, M-6ASA), iii) OPC with 2 %, 4 % and 6 % GGBFS replacement as part of cement (M-2GGBFS, M-4GGBFS, M-6GGBFS) and iv) OPC with 2 %, 4 % and 6 % ASA and 4 % GGBFS replacement as part of cement (M-2ASA 4GGBFS, M-6ASA 4GGBFS). Material preparation and casting procedures were done in accordance to British Standard (BS).

Physical testing including flow table test was carried out to determine workability of the mortar samples. After obtaining the optimum water to cement ratio, a new set of samples including cubes (compressive strength), cylinders (water absorption and porosity test) and prisms (flexural tensile strength and water absorption coefficient test) were casted to carry out respective testing. Besides that, initial setting time and final time were determined using vicat apparatus. The water curing method was used for further hydration of cement mortar. The samples were cured for 7 days and 28 days before undergoing compressive strength and flexural splitting test in order to determine the mechanical performance of ASA based mortar and to obtain the optimum ASA ratio as replacement of cement in mortar production. LCA of the possibility of ASA in cement production was carried out.

# **CHAPTER 2**

# LITERATURE REVIEW

# 2.1 Introduction

Mortar is a product of ordinary portland cement (OPC), sand and water. Mortar is commonly used as brick laying binding agent, plastering works to hide the joints to improve the appearance and provides an even bed to stones, bricks or concrete blocks to prevent their inequalities from bearing upon one another. By introducing ASA into mortar, the amount of cement required during the production can be reduced. Currently, the most common methods of sludge disposal are landfills or for agricultural purposes.

In recent years, studies have been carried out by various researchers regarding the use of sludge as a construction material. Other than using ASA in cement production, ASA has been used in mortars (Maha et al., 2011) in concrete mixtures (Haider et al., 2018) and in brick manufacture (Tay et al., 1987). The reuse of waste in cement production mainly depends on the chemical composition of the waste. ASA is one of the potential by-products from water treatment plant that possesses high content of SiO<sub>2</sub>, CaO, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. Burning ASA produces compounds such as calcium aluminates and calcium silicates which are commonly present in OPC (Haider et al., 2018).

The success in production of ASA cement based mortar not only can solve the landfill sites problem, but also reduces the dependent on raw material and enhance mortar properties in terms of strength, workability and durability. It has been expected greatly in the conservation of energy and environment by virtue of decreasing energy for production, reducing carbon dioxide emission, natural resources for cement and load to disposal sites (Lin, 2005).

# 2.2 Overview of Material

OPC, also classified as CEM I cement according to BS EN 197-1:2000 is the most common and widely use cement in construction activity. GGBFS is a by-product from steel and iron industry while ASA is a by-product from water treatment plant.

# 2.2.1 Chemical Composition of OPC, AS and GGBFS

In general, the chemical compositions of OPC are inconsistent due to supply from different manufacturing sources. The major chemical compositions of OPC are limestone, alumina and silica. They play a significant role in hydration process to form calcium silicate hydrate gel that contributes to the compressive strength on mortar. The findings of Haider showed that the chemical compositions of OPC, ASA and GGBFS varies from each other but all the major chemical compositions of OPC are present in both AS and GGBFS. The chemical composites of OPC, ASA and GGBFS are presented in Table 2.1 (Haider et al., 2014).

Chemical	Portland Comont	Alum Sludgo	Ground Granulated		
Composition (%)	Portiand Cement Alum Sludge	Blast Furnace Slag			
Silicon dioxide	20.19	42.29	22.00		
(SiO <sub>2</sub> )	20.18	42.38	32.00		
Aluminium trioxide	5.23	25.02	10.57		
(Al <sub>2</sub> O <sub>3</sub> )	5.23	35.03	12.37		
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	3.34	4.94	0.24		
Calcium oxide	64.40	0.13	41.0		
(CaO)	04.40	0.15	41.0		
Magnesium oxide	1.8	0.29	6.04		
(MgO)	1.8	0.29	0.04		
Sodium oxide	0.07	0.10	0.39		
(Na <sub>2</sub> O)	0.07	0.10	0.37		
Compound composition					
Tricalcium silicate	61.80				
(C <sub>3</sub> S)	01.80	-	-		
Dicalcium silicate	11.60	-	-		
(C <sub>2</sub> S)					
Tricalcium	° 20				
aluminate (C <sub>3</sub> A)	8.20	-	-		
Tertacalcium					
aluminoferrite	10.20	-	-		
$(C_4AF)$					

Table 2.1: Chemical Composition of Cement, Alum Sludge and GroundGranulated Blast Furnace Slag (Haider et al., 2014)

## 2.2.2 Pozzolanic Material

As stated in BS-EN 197-1:2000, pozzolanic materials which generally incorporated into cement are natural pozzolan and industrial by-products such as fly ash, silica fume and blast furnace slag. When clay minerals are calcined at temperatures between 600 °C and 900 °C, its components which mainly composed of  $C_3S$  and  $C_3A$  become highly reactive (Emad. Benhelal et al., 2013). The loss of water due to thermal treatments distorts the crystalline structure of the compound, thereby converts the compound into an unstable amorphous state. Pozzolanic material itself possesses low cementitious value but in the presence of water, it will chemically react with calcium hydroxide to form compounds that possess cementitious properties.

AS contains high percentage of silicon dioxide (42.38 %) followed by aluminium trioxide (35.03 %). The silicon dioxide in amorphous form will chemically react with calcium hydroxide produced from hydration process and lead to the production of calcium silicate hydrate (C-S-H) compound (Haider et al., 2018).

$$2S + 3CH \rightarrow C_3S_2H_3 \tag{2.1}$$

#### 2.2.3 Chemical Properties of Alum Sludge

Generally, the chemical compositions of AS are varies due to supply from different water treatment plants. Though, silica is still the major chemical composition in AS. The chemical composition of different AS used in several research studies are shown in Table 2.2.

Chemical	Wang	Haider	Frías
Composition			
Silicon dioxide	40.60	12 38	36.24
(SiO <sub>2</sub> )	40.00	42.36	50.24
Aluminium trioxide	41.31	35.03	29.46
(Al <sub>2</sub> O <sub>3</sub> )			
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	8.56	4.94	10.05
Calcium oxide	1.55	0.13	0.98
(CaO)			
Magnesium oxide	0.86	0.29	1.23
(MgO)			
Sodium oxide	0.18	0.10	0.83
(Na <sub>2</sub> O)			

Table 2.2: Chemical Composition of Alum Sludge Used In Several Researches(Wang, 2018; Haider et al., 2014; Frías, 2013)

# 2.2.4 Ground Granulated Blast Furnace Slag

Ground granulated blast furnace slag (GGBFS) is a by-product of iron and steel industry. GGFBS is obtained by quenching molten iron blast furnace slag in water to produce a glassy granular product. It is then dried and ground into fine powder. GGBFS has very similar chemical compositions to OPC such as 30-42 % of CaO, 35-39 % of SiO<sub>2</sub>, 10-14 % of Al<sub>2</sub>O<sub>3</sub> and 8-9 % of MgO (Siddique, R & Bennacer, R, 2012). GGBFS is a hydraulic material, which means that it will set and harden due to its chemical reaction with water. Concrete containing GGBFS cement has a higher ultimate strength than concrete that uses only OPC. When mixed with cement, GGBFS reacts with calcium hydroxide produced from clinker hydration to form additional hydrated calcium silicate. A more refined pores of cement matrix increase the chemical resistance of concrete (Siddique, R & Bennacer, R, 2012).

# 2.3 Setting Time of Mortar Incorporated with Alum Sludge

Setting time is important for purposes like handling, transporting, placing and giving required shape to mortar. Initial setting time is the time when the paste starts losing its plasticity. While the final setting time is the time between when water added to cement till it has come in hardened state. It is important to have sufficient setting time for transportation, placing and compaction of cement. Cement should neither set too rapidly nor too slowly. Rapid setting result in inadequate time to transport and work before it becomes too hard. Whereas, slow setting time will lead to delay in work.

According to Wang, increasing AS replacement ratio would delay setting time of mortar intensely. Incorporation of 12.5 % sludge would extend the final setting time from 19.3 hours to 29.8 hours, which exceeded the minimal requirement of 24 hours (Wang et al., 2015). The delay in setting time is due to the high content of clay with high water absorption properties. The presence of organic matter in sludge also hindered the hydration of cement which hampered the formation of calcium hydroxide. As a result, the setting time of mortar was considerably delayed.



Figure 2.1: Stiffening Time of Mortar at Varying Alum Sludge Replacement (Wang et al., 2015)

#### 2.4 Workability

Water is added to mortar to ease workability in construction work. Although addition of extra water increases the workability of mortar but this results in high porosity, which degrades durability and strength performances. The study carried out by Haider showed a decline in the slump of mortars with the increase in the amount of AS added (Haider et al., 2018). This behaviour is attributed to the rough texture of the ash particles which favours the adsorption of water.

### 2.5 Durability of Mortar Incorporated with Alum Sludge

The durability of masonry mortar is an important property that can significantly influence the performance of a masonry structure. Mortar with high porosity is more susceptible to chemical attack and weathering effect (Kim et al., 2014). There are a lot of factors affecting durability properties of mortar. Specific testing method is designated for each factor. In the research, durability of mortar is accessed in terms of its water absorption coefficient, porosity and water adsorption of the samples.

According to Haider, water absorption of composite cement with ASA and supplementary cementitious materials have low absorption on day 28, 56 and 90. The total porosity results of binary and ternary blends of cement decreased with an increase in age. The water absorption and porosity results of ternary blend cement outperform binary blend cement (Haider et al., 2018). This result may be due to pore refinement through filling and the secondary hydration reaction of the supplementary cementitious material. Pore refinement results in lower capillarity that contributes to higher durability properties of mortar (Siddique & Bennacer, 2012).

# 2.5.1 Water Absorption Coefficient

The water absorption coefficient can be determined by measuring the capillary rise absorption rate of mortar. It aims to characterise the tendency of a porous material to absorb and transmit water through capillary action. Water absorption coefficient illustrates the water mass uptake by concrete from the bottom surface. The lower the water absorption coefficient value is, the higher the water resistance of the mortar. A low water absorption coefficient value that was below 0.1 mm<sup>3</sup> /mm<sup>2</sup> /min<sup>0.5</sup> was expected in binary and ternary blend cement cast since low water to cement ratio is adopted (Haider et al., 2018). The decrease in water absorption coefficient was due to the increased density of the concrete, which led to finer pores and a smaller interconnected network of capillary pores.

# 2.5.2 Porosity

It is known that the use of pozzolans such as silica fume, GGBS, metakaolin and fly ash can improve the durability performance of concrete. The fine particles of pozzolanic materials can act as filler to densify the transition zone and reduce the porosity and permeability of mortar. A mortar with low porosity and permeability will resist undesirable phenomena. Concrete containing 15 % ASA has lower porosity compared to control concrete at all ages. The reduction in porosity of the ASA concrete probably due to the pozzolanic reaction and the filler effect of the ASA particles (Haider et al., 2018).

## 2.5.3 Water Absorption

Water absorption determines the amount of water absorbed by mortar, which indicates its degree of porosity, permeable pore volume as well as connectivity among these pores. When porosity decreases, there is a reduction in water absorption. It has been reported that the rate of water absorption of blended binder falls within the range of 36 % (Schutter et al., 2003). In general, multi-blended binders portray low absorption characteristics. This outcome is in agreement with the results reported by Haider, who noticed that mortar with different percentages of AS as the replacement of cement had absorption less than 10 % (Haider et al., 2018).

#### 2.6 Strength Development of Mortar Incorporated with Alum Sludge

At early stage, mortar incorporated with AS tends to show slower strength gain compared to that of OPC mortar. This is due to the pozzolanic characteristics possessed by AS, which extend the hydration process. Above all, organic matter will react with calcium ion  $Ca^{2+}$  complex and form a coating layer, which substantially retards hydration of cement (Wang et al., 2015). In the study conducted by Garces, 70 % cement with 30 % alum sludge in sample, silicate hydration was perceptibly retarded, possibly due to a reaction between the presence of fatty acids in organic matter and the  $Ca^{2+}$  and  $OH^-$  ions in the cement (Garces et al., 2008).



Figure 2.2: Compressive Strength for Ordinary Portland Cement: Alum Sludge Mixes (Wang et al., 2015)

#### 2.7 Relationship between Compressive Strength and Setting Time

Compressive strength is the most important mechanical property of every mortar as it plays a very important role in construction in order to sustain load. There are several factors that will affect the compression strength of mortar such as water-to-cement ratio, heat of hydration, set times and fineness of cement (Admed et al., 2008). The study carried out by Frías showed that every 2 % addition of AS increased water to cement ratio by 0.01 due to small particle size of AS. It was reported to delay the cement hydration and reduce the compressive strength (Frías et al., 2014).

## 2.8 Correlation between Compressive Strength and Flexural Strength

According to Wang, the compressive strength and flexural strength of the AS mortars declined significantly, even when only 12.5 % of the cement was replaced with AS. The 28 days compressive strength and flexural strength of AS mortar declined with increasing proportions of sludge (Wang et al., 2018). Similar result was obtained by Garces, whereby mortars containing sludge ash blended cements yielded lower compressive strength and flexural strength values than those of mortars with non-blended cements for each curing time (Garces et al., 2008). Both researchers concluded that the compressive strength of mortar has direct relationship with flexural strength of mortar.

# 2.9 Relationship between Strength and Durability

The durability of mortar against degradation is related to its characteristics of pore system which is measured in terms of permeability (Gambhir, 2013; Schutter & Audenaert, 2004). As cited from Audenaert, et al. (2004), the more porous the concrete, the material is more susceptible to degradation mechanism caused by penetrating substances such as water. Concretes with low porosity and permeability will resist undesirable phenomena like sulphate attack, acid attack and weathering. From Figure

2.3, strength is inversely proportional to capillary porosity. As the strength of concrete increases due to hydration, thus the permeability reduces significantly indicating a denser microstructure. Due to capillary pores are larger in size than gel pores, the permeability of cement paste is governed by the capillary porosity as shown in Figure 2.3 (Gambhir, 2013).



Figure 2.3: The Relationship between Compressive Strength and Porosity; & Permeability and Porosity (Gambhir, 2013)

#### 2.10 Summary

The use of pozzolanic materials from industrial by-products that can be utilised as replacements for cement has received considerable attention due to the benefits they bring in enhancing the mechanical and chemical properties of mortar. The study carried out by a few researchers concluded that addition of clay-rich AS in mortar dramatically increased water demand, resulting in longer setting time and lower compressive strength. High content of organic matter in AS significantly delays the hydration of cement. Hence, GGBFS is used as supplementary cementitious material in hope to effectively reduce water demand and provide moderate compressive strength. Finally, the durability of mortar mixes under varying environmental conditions was evaluated.
This chapter discussed the literature review on the past researches transforming AS into construction material. The area of concern for this study focuses on the mechanical properties and durability properties of mortars incorporated with ASA. It is believed that incorporation of ASA may result in increased value of strength properties and durability properties.

## **CHAPTER 3**

### METHODOLOGY

## 3.1 Introduction

This chapter describes the material used, the mixing procedures and the test procedures for the mortar specimens incorporated with ASA.

## **3.2 Raw Materials**

The sample production for mortar incorporated with AS consists of five types of raw materials, namely, OPC, ASA, sand, water and GGBFS.

# 3.2.1 Ordinary Portland Cement

The OPC which is produced by Hume Cement under the brand name of "Panda" was used throughout the study. According to BS EN 197-3, the OPC used throughout this research complies with CEM I Portland Cement and the detailed chemical composition of OPC is given in Table 3.1.

Chemical Composition (%)					Physical Properties		
CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	Na <sub>2</sub> O	Specific gravity	Fineness (m <sup>2</sup> /kg)
67.17	20.99	4.6	4.44	2.53	0.03	3.12	328

**Table 3.1: Chemical Composition and Physical Properties of the Cement** 



**Figure 3.1: Ordinary Portland Cement** 

# 3.2.2 Alum Sludge Ash

The AS was obtained from a private company water treatment works in Malaysia. The AS cake mainly consisted of 70 % moisture content and dried AS contained 15 % total organic carbon and 15 % of aluminium sulphate. The collected AS was oven dried at temperature of 105 °C  $\pm$  5 °C for 24 hours in order to remove the moisture content. Next, the AS was blended and sieved. The dried AS was sieved through a 600 µm sieve collected and stored in an airtight container.



(a) Alum sludge cake

(b) Ground alum sludge

Figure 3.2: (a) Alum Sludge Cake Before Oven Dried at 100 °C  $\pm$  5°C and (b) Ground Alum Sludge after Sieve Analysis

#### 3.2.3 Sand

In this study, only fine aggregate was used in the production of AS mortar production. Fine aggregate means the aggregate which passes through a 4.75 mm sieve. The sand was sieved through a 4.75 mm sieve before it was stored in a container. The sieving method of sand was either by hand or mechanically as described in BS EN 998-2.

#### 3.2.4 Water

In this study, tap water was used in the production of mortar. The water needs to be free from impurities and maintain a neutral pH, else the impurities may affect the process of hydration of cement and durability of mortar. Water was used in three parts of preparing the specimen in this study, which were (1) mixing AS for the production on mortar, (2) curing process and (3) medium for absorption testing.

#### 3.2.5 Ground Granulated Blast Furnace Slag

GGBFS was oven dried at temperature of 105 °C  $\pm$  5 °C for 24 hours in order to remove the moisture content. Next, the dried GGBFS was sieved through a 600 µm sieve, collected and stored in an airtight container. Table 3.2 shows that Silica (SiO<sub>2</sub>) is the most abundant chemical component in GGBFS, while Figure 3.3 shows the GGBFS after sieved.

Chemical Composition (%)					
CaCO <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	K <sub>2</sub> O	Na <sub>2</sub> O
15.54	82.26	1.29	0.31	0.14	0.03

Table 3.2: Chemical Composition of GGBFS



**Figure 3.3: Sieved GGBFS** 

### 3.3 Mould

In this study, three types of moulds were used to cast different types of mortar specimens. Mortar mixing, moulding and compaction were carried out in accordance to the BS EN 998-2. Table 3.3 shows the type, dimension and quantity of moulds required for each testing. Before fresh mortar paste was placed into the mould, the

mould was cleaned with no residue, all bolts that hold the mould were tightened to avoid leakage and lastly a layer of oil was coated on the moulds to provide ease of demoulding.

Testing Method	Type of Mould	Dimension	Number of	
			Mould Prepared	
Compressive	Cubic mould	50 mm $\times$ 50 mm $\times$	6	
strength test	Cubic mound	50 mm	0	
Porosity test and water absorption test	Cylindrical mould	Ø 45 mm × 40 mm	6	
Flexural strength test and water absorption coefficient	Prism mould	40 mm × 40 mm × 160 mm	6	

Table 3.3: Type, Dimension and Quantity of Mould Required

### 3.4 Mix Proportion

Trial mixes are not required in this study due to the variation in supplementary cementitious materials (SCM) content in the specimens. The mixture design for mortar production from AS was classified into four groups (Table 4.1). The cement to sand ratio was kept at 1:3, while water to cement ratio was fixed at 0.60. The adequate proportion of sludge as cement substitute (0, 2, 4, and 6 % replacement of cement by sludge) was determined first. Next, the effectiveness of varying GGBFS content was evaluated. Finally, the alum sludge content increased from 2 % to 6 % and GGBFS was fixed at 4 %. The production of mortar involved mixing, flowability determination, casting, and curing phases.



Figure 3.4: Materials Included in Mortar Production

# 3.5 Mixing Procedure

The mixing procedure in this study was carried out in accordance to the BS EN 998-2, all the mixings were carried out manually. OPC, sand, ASA, GGBFS and water were weighed for preparation of raw materials. First, the dry mix was produced by manually mixing dry material in a mixing bucket. Water was then added into the dry mix. The mixture was mixed uniformly followed by flow table spread test. Lastly, fresh mortar was poured into the mould.

#### 3.6 Curing

Curing condition is very important in gaining the strength of mortar. Curing of mortar specimen is commenced as soon as after adequate hardening of the sample under room temperature for minimum of 18-24 hours. For this study, mortar specimens were cured in a water tank for 7 days and 28 days until age of testing.

#### 3.7 Fresh Cement Mortar Testing Method

There are two fresh cement mortar tests carried out in this study, namely flow table test and setting time test.

#### 3.7.1 Flow Table Spread Test (BS EN 1015-3)

This test is performed to determine the consistency and workability of the fresh mortar. This test is in compliance to the specification in BS EN 1015-3. In this study, mortar was poured into the conical mould that was placed at the centre of flat surface as shown in Figure 3.5. The conical mould was then removed following by 15 drops. The diameter of the mortar flow was measured in orthogonal directions to determine the consistency of mixes.



Figure 3.5: Set Up of Flow Table Spread Test

## 3.7.2 Initial Setting Time and Final Setting Time (BS EN 196-3)

This test is to determine the setting time of cement by vicat apparatus in accordance to BS EN 196-3. It is essential that the cement sets neither too rapidly nor too slowly.

300 g of cement and 180 ml of water were mixed in a mixing bowl. Next, the cement paste was filled into the vicat mould. A square needle of cross section 1 mm x 1 mm was released to penetrate into the cement paste. This step was repeated at regular intervals until the needle failed to penetrate 5 mm measured from the bottom of vicat mould. The time was recorded as initial setting time. Next, a needle with annular collar was attached to the moving rod. The final setting time was recorded when outer needle leaves no impression on the cement surface. Figure 3.6 shows the surface of sample after conducting the test using vicat apparatus.





(a) Surface of sample indicating final setting time

(b) Vicat apparatus

Figure 3.6: The Apparatus Set Up of Vicat Test and Surface of Sample after Conducting Test

# 3.8 Attenuated Total Reflectance-Fourier Transform Infrared Spectrometry (ATR-FTIR)

ATR-FTIR is a useful tool in determining the IR spectrum of samples. This technique measures the absorption of infrared radiation by the sample material versus wavelength. A small amount on sample is placed at the centre of optically dense crystal with a high refractive index. After each successive test, the surface of the ATR crystal

was cleaned using cotton containing isopropanol in one direction. The intensity can be plotted as the percentage of light transmittance or absorbance at each wavenumber. The functional group of ASA and GGBFS are interpreted through the generated IR spectrum. The purpose of test is to identify the presence of organic and in some cases inorganic compounds.



Figure 3.7: Attenuated Total Reflectance-Fourier Transform Infrared Spectrometry (ATR-FTIR)

# **3.9** Mechanical Test

There are two destructive harden mortar tests carried out in this study, namely compressive strength test and flexural strength test.

#### **3.9.1** Compressive Strength Test (BS EN 12390-3)

The compressive strength test was conducted in accordance to BS EN 12390-3, (2002) using the Compressive Testing Machine. Three cubic specimens with dimension of 50

 $mm \times 50 mm \times 50 mm$  were tested and average values of the readings were obtained for each batch of mix. The dimension of the specimen was measured by using vernier calliper to determine the cross-sectional area before being tested. Then, the specimen was placed at the centre of testing machine as shown in Figure 3.8. The test was then started as the specified loading rate until the test specimen failed. The maximum load attained was recorded in order to determine the compressive strength. The mean value of compressive strength obtained from three cubes was then taken as cube compressive strength for each mortar mix. The compressive strength of the specimen was calculated by using Equation 3.1.

$$f_c = \frac{P}{A} \tag{3.1}$$

where,

 $f_c = compressive strength, MPa$ 

P = maximum load sustained by specimen, N

A = cross-sectional area of specimen which load applied,  $mm^2$ 



Figure 3.8: Set Up of Apparatus for the Compressive Strength Test

#### **3.9.2** Flexural Strength Test (BS EN 1015-11)

Flexural strength test was performed with centre point loading method in accordance to BS EN 1015-11. Three prismatic specimens with dimension of 40 mm  $\times$  40 mm  $\times$ 160 mm were used in this test as shown in Figure 3.9. A 20 mm offset from the end of both the sides of prism was marked and placed on the support block. The specimen was loaded gradually with a constant rate of loading until failure. The maximum load attained was recorded in order to determine the flexural strength. The mean value of flexural strength obtained from three prism was then taken as flexural strength for each mortar mix. The flexural strength of the specimen was calculated by using Equation 3.2.

$$R = \frac{3Pl}{2bh^3}$$
(3.2)

where,

R = flexural strength, MPa

P = maximum load applied, N

l = length of specimen, mm

b = width of specimen, mm

h = depth of specimen, mm



Figure 3.9: Schematic Diagram of Flexural Strength Test Set Up

The durability test was conducted to determine the performance of mortar under different exposed conditions. The test included water absorption coefficient test, porosity test and water absorption test.

#### 3.10.1 Water Absorption Coefficient Test (BS EN 1015-18)

Water absorption coefficient test was conducted in accordance to BS EN 1015-18 (2002). In this study, the prism specimens (40 mm  $\times$  40 mm  $\times$  160 mm) were oven dried for 24 hours at 60 °C ± 5 °C. The weight of the oven dried specimens were recorded to the nearest 0.01 g. Waterproof tape was used to prevent side absorption and to ensure unidirectional flow. During testing, the bottom of the specimen were immersed in a tray of water to a maximum depth of 5-10 mm by resting on steel rods to permit free water movement as shown in Figure 3.10. The uptake of water by capillary absorption was measured through mass of the specimen at intervals of 5, 10, 15, 30, 60, 90, 120, and 150 minutes from the start of the test. After testing, the bottom surface where in contact with water was wiped off with a paper towel to remove any excess water. The weighing operation was completed within 30 seconds. After weighing, the specimen was returned to the tray immediately and proceeded until the end of the experiment. The coefficient of water absorption per unit area of the mortar at each time interval was calculated according to the Equation 3.3:

$$I = \frac{W_{w} - W_d}{W_d} \times 100 \tag{3.3}$$

where,

I =Rate of water adsorption, %

 $W_w$  = Weight of mortar after immersed in water, g

 $W_d = dry$  weight of mortar, g



Figure 3.10: Schematic Diagram of Water Absorption Coefficient

### 3.10.2 Porosity Test (BS EN 1881-122)

A measure of porosity provides an important indication and assessment on the durability of mortar. Porosity test was conducted in accordance to BS EN 1881-122. The testing apparatus is designed by applying the concept of Archimedes' principle, which measures the upward buoyant force that is exerted on the mortar specimen immersed in the water. In this study, the cylindrical specimens ( $\phi$ 44 mm × 40 mm) were taken out one day in advance from the curing tank. The specimens were wiped to surface dry condition and weighed to obtain the saturated surface dry weight, W<sub>sat</sub> of the specimens. Next, the specimens were submerged into the water buoyant apparatus as shown in Figure 3.11 where the weight of displaced water indicated by the buoyant balance was recorded as W<sub>wat</sub>. The specimens were oven dried for one day at 60 °C ±5 °C. The oven dried weight of specimens were recorded as W<sub>dry</sub>. The porosity of the mortar was calculated according to the Equation 3.4:

$$P_{\rm r} = \frac{W_{\rm sat} - W_{\rm dry}}{W_{\rm sat} - W_{\rm wat}} \times 100$$
(3.4)

where,

 $P_r = Porosity, \%$ 

 $W_{sat}$  = saturated surface dry weight of specimen, g

W<sub>dry</sub> = oven dried weight of specimen, g

 $W_{wat} = mass of sample in water, g$ 



Figure 3.11: Set Up of Water Buoyancy Apparatus

#### 3.10.3 Water Absorption Test

Water absorption test was carried out in this study to determine the water absorption capacity of the hardened mortar sample in accordance to BS 1881-122. Additionally, measuring absorption provides an understanding of the permeable pore volume and connectivity among these pores. The cylindrical specimens ( $\emptyset$ 44 mm × 40 mm) were wiped to surface dry condition and weighed to obtain the saturated surface dry weight,  $W_{sat}$  of the specimen. Next, the specimens were oven dried for 24 hours at 100 °C ± 5 °C. The weight of oven dried specimens,  $W_{dry}$  were obtained. The water absorption of mortar was calculated according to Equation 3.5:

W.A.=
$$\frac{W_{\text{sat}} - W_{\text{dry}}}{W_{\text{dry}}} \times 100\%$$
 (3.5)

where,

W.A. = water absorption of hardened mortar specimen, %  $W_{sat}$  = saturated surface dry weight of mortar specimen, kg  $W_{dry}$  = oven-dried weight of specimen, kg

#### 3.11 Life Cycle Assessment (LCA)

Life cycle assessment (LCA) is used to evaluate the environmental impacts of a product or process throughout its entire product life from the extraction of raw materials for manufacturing to the end user. The rate of carbon emission and total energy usage for production of ASA cement mortar (M-ASA GGBFS) in terms of embodied carbon (EC) and embodied energy (EE) are evaluated and compared with conventional cement mortar production.

## 3.12 Summary

Mortar incorporated with ASA and GGBFS as replacement of cement were produced. Several mix proportions were prepared in this study namely; M-CTR, M-2ASA, M-4ASA, M-6ASA, M-2GGBFS, M-4GGBFS, M-6GGBFS, M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS. Total of six cube samples, six prism samples and six cylinder samples for each mix proportions were produced. The specimens were cured under water curing conditions for 7 days and 28 days followed by the properties testing namely workability test, setting time test, compressive strength test, flexural strength test, water absorption coefficient test, porosity test and water absorption tests.

# **CHAPTER 4**

# WORKABILITY, SETTING TIME, ATR-FTIR, COMPRESSIVE STRENGTH AND FLEXURAL STRENGTH

# 4.1 Introduction

This chapter discusses the main results of workability, setting time, compressive strength and flexural strength that were carried out on mortar samples, namely M-CTR, M-2ASA, M-4ASA, M-6ASA, M-2GGBFS, M-4GGBFS, M-6GGBFS, M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS. Each of the mortar samples was water cured for 7 days and 28 days before the tests were carried out. The effect of ASA as cement replacement material on its physical properties (setting time and workability), chemical properties (functional group) and mechanical properties (compressive strength and flexural strength) are discussed in this chapter.

## 4.2 Mix Proportions

Table 4.1 presents the mix proportions used in this study for M-CTR, M-2ASA, M-4ASA, M-6ASA, M-2GGBFS, M-4GGBFS, M-6GGBFS, M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS.

Types of	Material (%)			Water	Binder	Density
Sample	Cement	ASA	GGBFS	/Cement	/Sand	$(\text{kg mm}^{-3})$
				Ratio	Ratio	
M-CTR	100	0	0	0.60	1:3	2072
M-2ASA	98	2	0	0.60	1:3	1928
M-4ASA	96	4	0	0.60	1:3	1760
M-6ASA	94	6	0	0.60	1:3	1600
M-2GGBFS	98	0	2	0.60	1:3	2008
M-4GGBFS	96	0	4	0.60	1:3	2032
M-6GGBFS	94	0	6	0.60	1:3	2080
M-2ASA	94	2	4	0.50	1.3	2024
4GGBFS	74	2		0.50	1.5	2024
M-4ASA	92	4	4	0.60	1.3	2096
4GGBFS	72			0.00	1.5	2070
M-6ASA	90	6	4	0.60	1.3	2120
4GGBFS	20	0		0.00	1.5	2120

**Table 4.1: Mix Proportions** 

Notes: CTR = Control mix; ASA = Alum sludge ash; GGBFS = Granulated ground blast furnace ash; W/C = water to cement ratio

The mixture design for mortar production from AS was classified into three phases. The adequate proportion of AS as partial replacement of cement was determined. The cement-to-fine aggregate ratio was kept at 1:3 and water to cement ratio was fixed at 0.60. Next, the optimum varying content of granulated ground blast furnace ash, GGBFS as supplementary cementitious materials, SCM was evaluated. Lastly, the effectiveness of varying ASA and fixed GGBFS was evaluated. The density of mortar reduced with the increase of ASA replacement with ordinary portland cement, OPC. The densities of the samples in the study ranged from 1440 kg m<sup>-3</sup> to 2120 kg m<sup>-3</sup>. M-6ASA mixture had the lowest density, because ASA has a much lower specific gravity than cement, thus reducing the mass per unit volume. M-6ASA achieved approximately 22 % density reduction, as compared to the M-CTR, as shown in Table 4.1.

#### 4.3 Workability and Setting Time

The tests on physical properties of incorporation of ASA and GGBFS in mortars were focused on workability and setting times. The workability result for all samples are illustrated in Figure 4.1.



Figure 4.1: Workability for Incorporation of ASA and GGBFS in Mortars

The workability of mortar decrease with the increase of the ASA and GGBFS mortar ratio blended in the cements. This phenomenon could mainly result from the high water absorption of AS due to high content of clay mineral illite–montmorillonite as confirmed by ATR-FTIR testing. Given that water to cement ratio is fixed, the increase of ASA content with high water absorption properties in cement trap large portion of water in its pores lead to decrease in workability.

A similar study on AS as cement replacement in mortar by Frías, opined that the higher fineness of the AS than the OPC and to the clayey nature of the waste would cause strong water retention powers to result in higher water requirement (Fraís et. al, 2014). All the results obtained showed that the workability was 120 mm  $\pm$  10 mm. The results were above of the minimum workability required by the standard specification BS EN 1996-1-1: 2005.



Figure 4.2: Setting Time for Incorporation of ASA and GGBFS in Mortars

Figure 4.2 illustrates that the initial setting time and final setting time of mortar dramatically extended with increasing AS replacement ratio as compared to control mix. The M-6ASA sludge incorporation lengthened the final setting time from 6.3 hours to 36 hours. This phenomenon could mainly result from the presence of fatty acid inside the ASA. The deposit of ASA of surface of cement result in low heat of hydration at early stage (Albayrak et al., 2005). As a result, the setting time of M-2ASA, M-4ASA and M-6ASA are significantly delayed.

According to Halaweh, the presence of sulphate ions interrupted cement hydration, because it would hamper the calcium hydroxide formation. Sulphates are known to retard the setting time of cement mortar or concrete. Tricalcium aluminate,  $C_3A$  is responsible for early strength of cement. Sulphates reacts with  $C_3A$  in cement with the presence of water to form ettringite. Ettringite slows down the hydration process by forming a diffusion barrier on the surface of  $C_3A$ . The presence of sulphate ions also contributes to the formation of excess gypsum in the concrete which slows down the hydration process and thereby retarding the setting of the cement mortar or concrete (Halaweh et.al, 2006).

Studies showed that GGBFS contains high content of calcium and performs as a hydraulic material (Cheah et. al, 2016). The formation of calcium hydroxide during spontaneous hydraulic reaction may relieve the delayed effect by sulphate ion in AS. Figure 4.2 shows the initial setting time of M-2GGBFS, M-4GGBFS and M-6GGBFS was 10 %, 30 % and 35 % higher than that of M-CTR, respectively. Similar result was reported by Wainwright and Ait-Aider, where increase in content of GGBFS in cement led to delay in setting time (Wainwright & Rey, 2000). Meanwhile, ternary cement with ASA and GGBFS mixed exhibit an increase in initial and final setting time of M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS but well below compared with M-2ASA, M-4ASA and M-6ASA. This shows that the GGBFS has compensated the setting time of cement.

# 4.4 Analysis of Raw Materials using Attenuated Total Reflectance-Fourier Transform Infrared Spectrometry (ATR-FTIR)

For this study, ASA and GGBFS were used as partial replacement of cement in mortar production. According to Wang, ASA consists abundant of organic matter which would hamper strength development (Wang et al., 2018). The ATR-FTIR is used to investigate the functional groups present in both the binders so that it is easier to understand the chemical reactions in the production of cement mortar. Figure 4.3 shows the ATR-FTIR result of ASA. As for GGBFS, it is also used as one of the material in the production of cement mortar. Therefore it has also undergone the ATR-FTIR analysis to identify its possible mineral present which is shown in Figure 4.4.

# 4.4.1 Analysis of ASA using Attenuated Total Reflectance-Fourier Transform Infrared Spectrometry (ATR-FTIR)



The ATR-FTIR Spectrum of ASA is illustrated in Figure 4.3.

Figure 4.3: ATR-FTIR Spectrum of ASA

From Figure 4.3, it can be seen that there is a broad peak at 3380.96 cm<sup>-1</sup> which indicates the existence of carboxylic acid functional group due to the presence of high percentage of fatty acid in the ASA (Rodriguez et al., 2009). Fatty acids are made up of long chain carbon skeleton, which it indicates the presence of high content of organic matter inside ASA that will cause mortar to develop lower compressive strength. Fatty acids contribute to hydrophobic effect and result in low water absorption and high water repellence. Other than that, alkane compound has also contributed to (C-H) at 2922.53 cm<sup>-1</sup> and 2853.8 cm<sup>-1</sup> which are contributed by those hydrocarbon components. Small peaks at 1728.53 cm<sup>-1</sup> and 1632.95 cm<sup>-1</sup> show that there are the existence of (C=O) compound of ester. The bands at 1460.04 cm<sup>-1</sup> and 1101.15 cm<sup>-1</sup> are attributed to the stretching vibrations from the Si-O bonds in the quartz. At 529.86 cm<sup>-1</sup> shows strong (C-H) bending by alkene compound. The intense band peaking at around 1039.48 cm<sup>-1</sup> along with the intense bands at 529.86 cm<sup>-1</sup> and the above-mentioned bands at 3380.96 cm<sup>-1</sup>, were assigned to illite–montmorillonite (Rodriguez et al., 2009).

# 4.4.2 Analysis of GGBFS using Attenuated Total Reflectance-Fourier Transform Infrared Spectrometry (ATR-FTIR)

The ATR-FTIR Spectrum of GGBFS is illustrated in Figure 4.4.



Figure 4.4: ATR-FTIR Spectrum of GGBFS

From the results, it can be observed that at peak  $1431.00 \text{ cm}^{-1}$ , there are sulphate ester (S=O) contributed by miscellaneous compounds. The band at 874 cm<sup>-1</sup> shows a hydrocarbon (C-H) bond contributed by alkane compounds.

#### 4.4.3 Chemical Reaction

Two major compounds in portland cement, tricalcium silicate (C<sub>3</sub>S) and dicalcium silicate (C<sub>2</sub>S) contribute to the strength of the finished mortar. When water is added to the cement, these compounds undergo hydration. The reaction releases  $Ca^{2+}$ , OH<sup>-</sup> ions and a large amount of heat. Possible reactions when ASA mix with cement include:







Figure 4.5: (a) Chemical Structure of Stearic Acid Salts  $Mn^+ = Al^{3+}$  and (b) Chemical Reaction between Stearic Acid on Clinker

clinker surfaces

Metallic soaps are salts with long chain fatty acids. The long hydrophobic chains with water repelling property prevents penetration of water into cement mortar. Mortar for external use usually contain metallic soaps (stearates and oleates) to provide a protection from weathering effect (Falchi et al., 2015). Fatty acids are saturated monocarboxylic acids that have long carbon chains (at least ten carbons) e.g.  $CH_3(CH_2)_{14}COOH_(Falchi et al., 2015)$ . This is confirmed by the carboxylic acid bond found in the results generated by ATR-FTIR for ASA. Fatty acids and aluminium

hydroxide form soaps with hydrophobic properties. The adsorption of the long fatty acid chain of the stearates and the ion exchanges with clinker grains might generate a hydrophobic layer on the clinker surfaces (Falchi et al., 2015) which could inhibit normal mortar hydration. Furthermore, the presence of excess gypsum further led to delay in setting time, as gypsum is added to regulate setting time of cement. If it is not added, the cement will set immediately after mixing with water leaving no time for mortar placing.

#### 4.5 Compressive Strength

The compressive strength test results for all the samples are illustrated in Figure 4.6. The test was conducted on days 7 and 28 of curing periods.



Figure 4.6: Compressive Strength on Days 7 and 28 of Curing Periods for Incorporation of ASA and GGBFS in Mortars

From Figure 4.6, the trend of strength development increases throughout the curing periods for all samples. Generally, it is clearly shown that the compressive strength for ASA mortar decrease with the increase of ASA content. Only 2.0 % sludge

replacement, M-2ASA led to the 14.30 % reduction on day 7 of the compressive strength. A similar study deduced that 12.5 % ASA as partial replacement of cement in normal mortar has resulted in 75 % reduction in compressive strength as compared to control mix (Wang et. al, 2015). According to Wang, high content of organic matter in sludge (21.6 % of LOI) also interfered cement hydration, because it would hamper the CH formation and impeded further cement hydration. Overall, all the samples show breaking strength of above 12 MPa which fulfilled the minimum compressive strength required by the standard specification BS EN 1996-1-1: 2005 except for M-4ASA and M-6ASA.

The increase in partial replacement of cement with ASA led to considerable decrease of hydration rates, which would explain their lower 7 days strength. The substantial decline in mortar performance and the setting alterations may be related to the organic matter in the ASA. As ASA also contained a significant amount of organic matter or humic matter as shown by the total organic carbon (TOC) value of approximately 15 %. Replacement of 6 % of cement significantly change the initial hydration stages, lengthening the setting time by over 24 hours. In the M-6ASA sample hydration reaction was retarded, possibly due to possible reactions between the fatty acids and the Ca<sup>2+</sup> and OH<sup>-</sup> ions in the cement, which would inhibit portlandite nucleation (Rodríguez, 2009). According to Albayrack et al. (2005), the presence of fatty acids lead to intense alterations in initial ettringite formation, delay in setting time and lower compressive strength. These results proved that the ASA failed to behave like either an active addition or an inert substance.

Therefore, to enhance cement hydration and setting time of the mortar, SCM were added to produce sludge derived mortar. Wang et al. (2015) reported that partial replacement of fly ash in cement increased the chemical resistance of mortars when compare to plain portland cement. Similarly, partial replacements of GGFBS in cement has found to be advantageous for enhancing the resistance of mortar against sulfate attack. GGFBS reacts with calcium hydroxide Ca(OH)<sub>2</sub> to substantially reduce the presence of Ca<sup>2+</sup> and OH<sup>-</sup> ions in the concrete, leaving significantly less Ca(OH)<sub>2</sub> to react to form ettringite (Siddique and Khan, 2011).

The compressive strength for GGBFS mortars increases with increase of GGBFS content throughout the curing periods. The M-4GGBFS mortar posed the highest compressive strength throughout the whole curing periods as compared to M-2GGBFS and M-6GGBFS. Thus, 4.0 % GGBFS incorporated ASA and mortars was investigated in the following experiments. At the age of 28 days, M-4ASA 4GGBFS recorded a higher compressive strength as compared to M-2ASA 4GGBFS and M-6ASA 4GGBFS. The presence of GGBFS increases the cementing properties in the mortar blended cements. This is because GGBFS contains high content of calcium oxide CaO and performs as a hydraulic material that harden after the addition of water by chemical process in which the mineral structure of the binder changes (Siddique and Khan, 2011).

The decrease in the compressive strength of the ternary blends of ASA and GGBFS mortar mixtures M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS are approximately 26.19 %, 29.76 %, and 36.90 % after 28 days compared to the control mortar. It is evident that the mortar that contains GGBSF could be the catalyst that improves the compressive strength of mortar as well as compensate for the potential strength-reducing effect of ASA, achieving a comparable strength to that of the binary blend in M-2ASA, M-4ASA and M-6ASA after 28 days. The results are consistent with previous tests on the performance of the ternary blends of ASA and pozzolanic materials in terms of compressive strength (Haider et. al, 2018).

Types of	Average	Index	Average	Index
Sample	Compressive	Activity on 7	Compressive	Activity on
	Strength on	Days, %	Strength on	Day 28, %
	Day 7, MPa		Day 7 , MPa	
M-CTR	18.667 <u>+</u> 1.15	100.000	$28.000 \pm 2.00$	100.000
M-2ASA	16.000 <u>±</u> 0.00	85.699	17.667 $\pm 2.52$	63.095
M-4ASA	$2.000 \pm 0.00$	10.712	$3.000 \pm 0.00$	10.714
M-6ASA	$1.000 \pm 0.00$	5.356	$2.000 \pm 0.00$	7.143
M-2GGBFS	$12.000 \pm 1.00$	64.274	29.333 ±2.31	104.762
M-4GGBFS	14.667 ±1.15	78.557	$32.000 \pm 3.46$	114.286
M-6GGBFS	17.667 ±2.52	94.626	28.667 $\pm 4.16$	102.381
M-2ASA	15 333 +1 15	82 128	20.667 +3.06	73 810
4GGBFS	15.555 <u>-</u> 1.15	02.120	20.007 <u>1</u> 3.00	75.010
M-4ASA	15 333 +2 31	82 128	19.667 +2.52	70 238
4GGBFS	15.555 <u>-</u> 2.51	02.120	<u>17.007 <u>1</u>2.52</u>	70.230
M-6ASA	16,000 +1,00	85 699	17 667 +2 52	63 095
4GGBFS	<u>10.000 <u>1</u>1.00</u>	05.077	<u>17.007 <u>1</u>2.32</u>	05.075

Table 4.2: Index Activity of Compressive Strength on Days 7 and 28 forIncorporation of ASA and GGBFS in Mortars

# 4.6 Flexural Strength

Flexural strength test results for all the samples are illustrated in Figure 4.7. The test was conducted on days 7 and 28 of curing periods to determine the ductility of the mortars.



Figure 4.7: Flexural Strength on Days 7 and 28 of Curing Periods for Incorporation ASA and GGBFS in Mortars

Figure 4.7 shows that the flexural strength is directly proportional to the curing age for every mix proportion. For mortar containing ASA, the flexural strength decrease with increase of ASA. Figure 4.7 illustrates that the flexural strength of mortar has similar trend with its compressive strength. In addition, Figure 4.7 shows the ternary blended mortar with M-2ASA 4GGBFS has achieved the highest flexural strength. This result is because the pozzolanic properties of GGBFS in mortar facilitate cement hydration and shorten stiffening time where it enhances flexural load materials and of the ASA reducing crack propagation. No evidence of flexural strength decreasing with curing time was observed, which indicates that no expansion phenomena due to the presence of sulphate in AS in the mixtures took place during the study period. Le-Chatelier needles test can be carried out in future to confirm that no expansion on mortar occurred.

Types of	Average Flexural	Index	Average Flexural	Index
Sample	Strength on Day	Activity on	strength on Day	Activity on
	7 , MPa	Day 7 , %	28, MPa	Day 28, %
M-CTR	8.921 ±1.74	100.000	$10.511 \pm 0.65$	100.000
M-2ASA	8.146 <u>±</u> 0.77	91.312	$8.977 \pm 0.40$	85.410
M-4ASA	$2.609 \pm 0.26$	29.250	3.847 <u>±</u> 0.65	36.603
M-6ASA	$0.306 \pm 0.06$	3.435	$0.323 \pm 0.08$	3.074
M-2GGBFS	3.703 <u>+</u> 0.80	41.508	3.989 <u>+</u> 0.68	37.955
M-4GGBFS	$5.706 \pm 0.81$	63.966	$7.142 \pm 0.43$	67.950
M-6GGBFS	$8.256 \pm 0.52$	92.542	9.334 <u>+</u> 0.75	88.807
M-2ASA	3 341 +0 50	37 449	$3645 \pm 054$	34 683
4GGBFS	<u>5.511 <u>+</u>0.50</u>	57.115	<u></u>	2 11000
M-4ASA	3 189 +1 05	35 747	3416 +0.18	32,505
4GGBFS	<u></u>	33.111	<u></u>	52.000
M-6ASA	2.221 +0.81	24,895	$2.995 \pm 0.25$	28,501
4GGBFS	<u></u>	2	<u></u>	20.001

Table 4.3: Index Activity of Flexural Strength on Days 7 and 28 for Incorporationof ASA and GGBFS in Mortars

Figure 4.8 shows the effect of mortar incorporation with ASA and GGBFS on its correlation between compressive strength and flexural strength at different curing periods. The data are plotted at two respective curing periods. The compressive strength is said to be directly proportional to flexural strength, which gives a  $R^2$  value of 0.5709 as illustrated in Figure 4.8. A similar study by Rodriguez showed that an increase in AS content in the mortar reduces both days 2 and 28 flexural strength and compressive strength (Rodriguez, 2009).



Figure 4.8: Correlation between Compressive Strength and Flexural Strength for Incorporation of ASA and GGBFS in Mortars after 28 Days of Curing Periods

#### 4.7 Summary

The effects of incorporating ASA and GGBFS into mortars as partial replacement of cement were examined in terms of workability, setting time, compressive strength and flexural strength. The workability decreases with increase of ASA content inside the mortar while the setting time increase with increase of ASA content inside the mortar. Compressive strength and flexural strength are also decreased with the increase of ASA content. This may be due to the presence of sulphate ion and organic matter from alum sludge ash interfered the calcium hydroxide Ca(OH)<sub>2</sub> formation and impeded further cement hydration. This is confirmed by ATR-FTIR tests on ASA and GGBFS. Hence, to promote cement hydration and stiffening, supplementary cementitious materials, GGBFS are needed to produce mortar.

Next, the optimum composition of GGBFS was determined. From the result, the increase in content of GGBFS did not facilitate cement hydration, which could be attributed from the high water demand of GGBFS to maintain sufficient flowability. M-4GGBFS has achieved the highest compressive strength where 4 % is the optimum

composition for GGBFS incorporated in mortar as partial replacement of cement was used to blend with ASA to produce ternary cement.

From this study, M-2ASA 4GGBFS posed the optimum result in terms of workability, setting time, compressive strength and flexural strength. This is due to the calcium oxide derived from the GGBFS contributed to the strength development. While the compressive strength is directly proportional to flexural strength for mortar incorporated with 2.0 % to 6.0 % ASA as a partial replacement of cement.

## **CHAPTER 5**

# WATER ABSORPTION COEFFICIENT, POROSITY AND WATER ABSORPTION

# 5.1 Introduction

This chapter discusses on durability properties result that are carried out on ASA mortar. All specimens were water cured for 28 days prior to testing. The effects of ASA as cement replacement material on its durability properties including water absorption coefficient, porosity and water absorption test are discussed in this chapter.

### 5.2 Water Absorption Coefficient

Water absorption coefficient is the ability of water movement in pores through the mortar by capillary suction when it is exposed to ambient medium. The initial absorptions of M-CTR, M-2ASA and M-4ASA samples increase sharply between 0 s<sup>0.5</sup> and 7.75 s<sup>0.5</sup> while the secondary absorption of all specimens increase gradually after 7.75 s<sup>0.5</sup> which are illustrated in Figure 5.1. The slope that is defined as water absorption coefficient of M-2ASA, M-4ASA and M-6ASA are lower than that of M-CTR as shown in Figure 5.1. M-6ASA has the lowest water absorption coefficient among all the samples. As the amount of ASA as partial replacement of cement increases, the water absorption coefficient decreases. The decrease in water absorption

coefficient is due to the presence of ASA in the cement where it is insoluble in water, leading to finer pores and smaller interconnected network of capillary pores.



Figure 5.1: Effect of Incorporation of M-2ASA, M-4ASA and M-6ASA in Mortar on its Water Absorption Coefficient after 28 days of Curing Periods



Figure 5.2: Effect of Incorporation of M-2GGBFS, M-4GGBFS and M-6GGBFS in Mortar on its Water Absorption Coefficient after 28 Days of Curing Periods

From Figure 5.2, a similar trend can be observed where the initial absorptions of all samples increase sharply between 0 s<sup>0.5</sup> and 7.75 s<sup>0.5</sup> while the secondary absorption of all samples increase gradually after 7.75 s<sup>0.5</sup>. M-4GGBFS has shown the highest slope among all other samples. The slope that is defined as water absorption coefficient of M-2GGBFS, M-4GGBFS and M-6GGBFS are almost similar to M-CTR as shown in Figure 5.2.



Figure 5.3: Effect of Incorporation of M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS in Mortar on its Water Absorption Coefficient after 28 Days of Curing Periods

Figure 5.3 shows that there is no uptake of capillary action for first 24.5 s<sup>0.5</sup> for M-4ASA 4GGBFS and M-6ASA 4GGBFS where the initial absorption of all samples increases gradually after 24.5 s<sup>0.5</sup> while secondary absorption occurs after 60 s<sup>0.5</sup>. M-4ASA 4GGBFS has shown the lowest slope among all other specimens. The slope that is defined as water absorption coefficient of all samples are lower than that of M-CTR as shown in Table 5.1. The water absorption coefficient of M-4ASA 4GGBFS is 46 % lower than that of M-CTR and 3 % lower than that of M-2ASA 4GGBFS. In other words, the incorporation of ASA in mortar leads to gradual reduction in absorption by capillary action as compared to control mix. The connected pores of M-4ASA

4GGBFS are lesser than that of M-CTR. The water absorption coefficient of all samples are directly proportional to the square root of time. This finding was similar to the results with thermally activated ASA reported by Haider, where the inclusion of ASA in ternary blend cement mortar performed better in terms of water absorption coefficient than OPC and GGBFS alone (Haider et. al, 2018).

The capillary water absorption coefficients for all the mortars is shown in Figure 5.4. The reference levels (W1 and W2) are the requirements set by the EN 998-1 standard. The method proposed in the EN 1015-18 standard is used to give an idea of the waterproofing effectiveness of each mortars. The waterproofing effectiveness of ASA is excellent even at only 2 % dosage and the mortar attained W2 level of performance. The reduction is even obvious at higher dosage of 6 % replacement. While the water absorption coefficient of mortar containing GGBFS increases with time. The water absorption percentages for blended ASA and GGBFS mortar show highest efficiency in waterproof where there is an approximately 65 % to 68 % of reduction compared to M-CTR. Immersion of six waterproofing admixtures namely calcium stearate, zinc stearate, sodium oleate, silicones and hydrophobic polymer at dosages up to 2 wt.% of sample after 90 minutes showed a reduction in absorption by 57-86 % (Lanzon & Garcia, 2009).



Figure 5.4: Water Absorption Coefficients of Alum Sludge Ash and Ground Granulated Blast Furnace Slag in Mortar
Types of	Water	Linear Equation	$\mathbb{R}^2$	Percentage of
Sample	Absorption			Water Absorption
	Coefficient			Coefficient
	$(kg/m^2min^{0.5})$			Correspond to
				Control Mix on
				Day 28
M-CTR	0.0342	y = 0.0044x + 0.1397	0.8641	100
M-2ASA	0.0204	y = 0.0034x + 0.0999	0.8197	60
M-4ASA	0.0163	y = 0.0021x + 0.0242	0.7092	48
M-6ASA	0.0076	y = 0.0010x - 0.0184	0.7016	22
M-2GGBFS	0.0425	y = 0.0055x + 0.0973	0.8862	124
M-4GGBFS	0.0485	y = 0.0063x + 0.1034	0.9340	142
M-6GGBFS	0.0449	y = 0.0058x + 0.0796	0.8837	131
M-2ASA	0.0119	$y = 0.0015x \pm 0.0333$	0.7108	35
4GGBFS	0.0117	y = 0.0015X + 0.0555	0.7100	
M-4ASA	0.0109	y = 0.0014x - 0.0174	0.8281	32
4GGBFS	0.0109	y = 0.0011A 0.0171	0.0201	52
M-6ASA	0.0110	y = 0.0014x - 0.0138	0.6380	32
4GGBFS	0.0110	y = 0.001 m 0.0150	0.0500	52

Table 5.1: Effect of Incorporation of ASA and GGBFS in Mortars on its WaterAbsorption Coefficient after 28 Days of Curing Periods

## 5.3 Porosity

The porosity for ASA mortars is shown in Figure 5.5. The M-CTR has a higher porosity compared to the mortars incorporated with ASA. Their respective porosities are 19.44 % (M-2ASA), 42.86 % (M-4ASA) and 77.30 % (M-6ASA) lower than M-CTR as shown in Table 5.2. The porosity of ASA mortars might depend not only on the ASA composition, but also on its distribution inside the mortar matrix (Wittmann et al., 2011). A more plausible mechanism is that pore network may also has been disrupted when ASA filled some of the voids. ASA acts as a filler within the pore

structure where ASA arranges itself within the microstructure or bonds with the clinker in a way that produces hydrophobic surfaces within.



Figure 5.5: Effect of Incorporation of ASA and GGBFS in Mortar on its Porosity on Day 28 of Curing Periods

Table 5.2: Effect of Incorporation of ASA and GGBFS in Mortar	on its Porosity
after 28 Days of Curing Periods	

Type of Sample	Percentage of Porosity Correspond to
	Control Mix on Day 28
M-CTR	100.000
M-2ASA	119.441
M-4ASA	142.857
M-6ASA	177.300
M-2GGBFS	39.565
M-4GGBFS	66.423
M-6GGBFS	69.505
M-2ASA 4GGBFS	158.472
M-4ASA 4GGBFS	174.120
M-6ASA 4GGBFS	180.305

### 5.4 Water Absorption

The water absorption of mortars incorporated with ASA and GGBFS after 28 days of curing periods is illustrated in Figure 5.6.



Figure 5.6: Effect of Incorporation of ASA and GGBFS in Mortars on its Water Absorption after 28 Days of Curing Periods

The water absorption results for ASA mortar are shown in Figure 5.6. The effect of incorporation of ASA into mortar has decreased its water absorption as compared to that of control mix. The water absorption of M-2ASA, M-4ASA AND M-6ASA are 7 %, 24 % and 70 % lower that of M-CTR, respectively. However, water absorption of M-2GGBFS, M-4GGBFS and M-6GGBFS are 69 %, 38 % and 32 % higher than M-CTR, respectively as shown in Table 5.3. The incorporation of 6% of ASA as partial cement replacement material has outstanding effect on its water absorption. This result verifies the effect of ASA on lowering the porosity and connectivity among the pores inside mortar matric structure. Similar study conducted by Haider et al, (2018) opined that partial replacement with ASA with silica fume (SF), GGFBS and palm oil fuel ash (POFA) resulted in pore refinement and increased water resistance of the binder matrix.

As presented in Figure 5.6, the results show that the sample of ternary blended mortar possesses a lower water absorption rate compared to M-CTR. The water absorption for mortar specimens M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS at day 28 decrease by approximately 56 %, 71 % and 77 %, respectively as compared to M-CTR. The lowest level of water absorption is obtained from mortar with 6 % ASA replacement, with a value of 1.579 water absorption for the specimens on day 28. The results clearly prove that the use of ASA improves the characteristics of water absorption as compared to the binary blends with GGBFS for the same replacement level. Generally, mortar mixtures with ASA blended with GGBFS demonstrate low absorption characteristics. These findings are convincing, as the use of ASA would lead to an overall improvement in cement durability. A decrease in water absorption of ASA mortar leads to an increase in mortar resistance toward deterioration mechanisms that require water as the reaction medium. The restriction of water absorption is beneficial to the durability of mortar in term of decreasing penetration of chlorides and sulphates and reducing the amount of freezable water (Hong S. Wong et al., 2015).

 Table 5.3: Effect of Incorporation of ASA and GGBFS in Mortars on its Water

 Absorption after 28 Days of Curing Periods

Type of Sample	Percentage of Water Absorption		
	Correspond to Control Mix on Day 28		
M-CTR	100.000		
M-2ASA	92.123		
M-4ASA	75.516		
M-6ASA	29.213		
M-2GGBFS	169.532		
M-4GGBFS	138.396		
M-6GGBFS	132.652		
M-2ASA 4GGBFS	43.877		
M-4ASA 4GGBFS	28.246		
M-6ASA 4GGBFS	22.291		

### 5.5 Compressive Strength and Porosity Relationship

The relationship between compressive strength and porosity for mortars incorporated with 2 % to 6 % ASA as partial replacement of cements after 28 days of curing periods is illustrated in Figure 5.7.



Figure 5.7: Strength-Porosity Relationship for Mortar Incorporated ASA and GGBFS as Partial Cement Replacement Material after 28 Days of Curing Periods

Basically, there is a fundamental inverse relationship between porosity and compressive strength of mortar. In this study however, the compressive strength is found to increase with porosity, which gave a  $R^2$  value of 0.7009 as illustrated in Figure 5.7. A previous study has opined that calcium carbonate has the ability to enhance the strength by reducing its porosity (Haider et al., 2018). The contrary with previous result may be due to the presence of aluminium oxide inside AS that would react with calcium hydroxide, leading to displacement on calcium carbonate content. It can be deduced that the compressive strength is directly proportional to its porosity in this study.

In general, porosity would decrease with the increase of amount of ASA blended in mortar sample. Figure 5.7 shows that the lowest porosity values belonged to mortars with the highest replacement levels of ASA blended in mortar sample. This result is inspiring because it indicates that cement with ASA contributes to improvement in durability.

## 5.6 Summary

ASA in mortar as partial replacement of cement plays a significant role in enhancing its durability properties in terms of water absorption coefficient, porosity and water absorption. All M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS have lower water absorption coefficient as compared to the control mix. This is due to the presence of fatty acid that interrupts the capillary pores which decreases the ingress of water in the mortar. Low water absorption coefficient indicates a better durability and poor water absorption properties of mortar. All M-2ASA 4GGBFS, M-4ASA 4GGBFS and M-6ASA 4GGBFS have lower porosity and water absorption as compared to that of the control mix. The permeability of mortar is governed by the capillary porosity. The lower porosity will lead to lower permeability of water into mortar.

# **CHAPTER 6**

## LIFE CYCLE ASSESSMENT (LCA)

# 6.1 Introduction

Life cycle assessment (LCA), also known as "from cradle to grave analysis" is used to evaluate the environmental impacts of a product or process throughout its entire product life from the extraction of raw materials for manufacturing to the end user. The production of cement consumes huge amount of raw materials, energy and heat which lead to the production of significant amount of carbon dioxide. Therefore, LCA is used to assist in environmental management in sustainable development (Huntzinger et al., 2009). This includes the determination of rate of carbon emission and total energy usage for production of ASA cement mortar (M-ASA GGBFS) in terms of embodied carbon (EC) and embodied energy (EE) and comparison with conventional cement mortar production.

# 6.2 Embodied Carbon (EC) and Embodied Energy (EE)

Embodied carbon (EC) is defined as the total carbon footprint of the material from its raw materials extraction to its end of life, taking consideration of "cradle to gate", "cradle to site" and "cradle to grave" assessment. Cradle to gate assessments evaluate impacts of producing cement from the raw material extraction process to the packaging and shipping process. As for cradle to site means the transportation of the material to the site. Lastly, cradle to grave means the usage and maintenance of the product until the end of its life. Cradle to grave assessments are complicated especially for cement because cement has various end of life uses (Huntzinger et al., 2009). Therefore, the assessments reviewed here are "cradle-to-gate" studies omitting the end use and disposal of cement. Embodied energy (EE) is defined as the total energy required to produce the material.



Figure 6.1: Scope of Comparative LCA for Cement Manufacturing Process



Figure 6.2: Material Flow Diagram for the Production of 1000 kg of Ordinary Portland Cement (Huntzinger et al., 2009) and Composite Cement

Based on Figure 6.2, an average 1598 kg of raw materials are required to produce 1000 kg of finished cement. The yield of the end product is approximately 62.5 % for OPC and 69 % for blended cement. In other words, a substitution of 8 % of clinker needed to produce 1000 kg of cement is reduced from 950 kg to 870 kg.

Cradle to Gate – Materials Extraction and Manufacturing						
Materials Extraction Process						
Material	Material	Emission	Estimated	Transport	Total	
	required for	Factor, kg	Transportation	Emission,	Emission,	
	production of	$CO_2 / kg$	distance, km	kg CO <sub>2</sub> /km	kg CO <sub>2</sub>	
	1000 kg of					
	cement, kg					
ASA	40	0.00	100	0.0001	0.0100	
GGBFS	40	0.00	100	0.0001	0.0100	
Raw	1450 17	0.00	100	0.0001	0.0100	
Material	1430.17	0.00	100	0.0001	0.0100	
Manufactur	ring Process					
Operation	Energy	Electricity U	Electricity Usage, kWh Emission			
				Factor, kg	Emission,	
				CO <sub>2</sub> /kWh	kg CO <sub>2</sub>	
Crushing,						
grinding,	Electricity	80.66		0.32552	26.256	
Diending						
Pre- heating	II.	1174.04		0 22946	207 422	
and kiln	Heat	11/4.24		0.33840	397.433	
Finishing						
grinding,	Electricity	147.78		0.32552	48.105	
blending	-					
Total Carbon Emission per 1000 kg of cement, kg CO <sub>2</sub>					471.794	
Carbon Embodied, kg CO <sub>2</sub> /kg				0.471		

Table 6.1: Embodied Carbon of Cement containing ASA and GGBFS

\* Emission factor were obtained from Department for Business, Energy & Industrial Strategy, UK (BIES).

Notes: ASA: Alum sludge ash; GGBFS: Ground granulated blast furnace slag

Materials	Embodied	Material required for	Total
	Energy, MJ/kg	production of 1000 kg of	embodied
		mortar, kg	energy, MJ
ASA	n.d.	12	n.d.
GGBFS	0.61	12	7.32
OPC	4.60	276	1269.6
Sand	0.10	900	90
Water	0.20	180	36
Total Emission per 1000 kg of mortar, MJ			1402.92
Embodied Energy, MJ/kg			1.402

Table 6.2: Embodied Energy of M-ASA GGBFS

\* Emission factor were obtained from ICE Ver. 1.6a

Notes: ASA: Alum sludge ash; GGBFS: Ground granulated blast furnace slag; OPC: Ordinary Portland cement; n.d.: no data

The main GHG generated from the manufacturing of cement is carbon dioxide. Approximately half of the carbon released during the manufacturing of cement originates from the calcination process at 1500 °C. The remaining carbon is resulted from the burning of fuels to fire the kiln and powering during the manufacturing processes. The calculation for GHGs emission is based on Equation 6.1 as shown below. The emission conversion factor is based on the type of activities carried out by BEIS, 2018.

GHG emissions = activity data 
$$\times$$
 emission conversion factor (6.1)

Transportation distance for all the raw materials is from site to manufacturing plant based on average distance. The energy and heat input depend heavily on the manufacturing facility and a unique ratio of fuel types which including natural gas, fuel oil, tires and hazardous materials (van Oss & Padovani, 2002).

The EC of M-ASA GGBFS is 0.471 kg  $CO_2$  per kg which is extremely low compared to the conventional ordinary cement production. According to the International Energy Agency's (IEA) Greenhouse Gas R&D Programme, cement production generates an average world carbon emission of 0.81 kg  $CO_2$  per kg of cement produced (Hendriks et al., 2000). This is because the utilisation of waste materials reduces the amount of clinker used in cement production. The temperature required in calcination process is 1500 °C, while the temperature required to dry ASA is 100 °C. The EE of M-ASA GGBFS (1.402 MJ per kg) does not show significant difference compared to average EE of OPC (1.400 MJ per kg-1.500 MJ per kg) (ICE Ver. 1.6a). This is because EE found from GGBFS is 0.61 MJ/kg. Generous amount of energy is needed for production of GGBFS through the quenching molten blast furnace slag (a by-product of iron and steel making) in water to produce a glassy granular product that is then dried and ground into a fine powder GGBFS.

As ASA is a waste produced by water treatment plant and it is bearing a negative cost waste therefore ASA is not given an environmental benefit or penalty in LCA. In other words, ASA is considered as benign inputs. It is not associated with environmental impact. This is a reasonable assumption since ASA would be produced regardless of their inclusion in cement products. Hence, substitution of ASA would reduce the demand for clinker, which proportionately reduce the environmental impacts of the end cement product.

# 6.3 Economic Evaluation

Economic evaluation is also an important step that needs to be considered as it also serves as a criterion on becoming a feasible blended cement. The total costing per 1000 kg of mortar production is calculated in Table 6.3. The price rate for ASA is RM 0.00, as it is a negative cost waste, it has zero value. As compared to the ordinary mortar, the ASA GGBFS mortar reduces the cost by approximately 8 %.

Description	Rate	Input	Amount	Rate per	Amount	Rate per
	(RM)	power	used for	1000 kg of	used for	1000 kg of
		(kW)	production	M-ASA	production	ordinary
			of 1000 kg	GGBFS	of 1000 kg	mortar
			mortar	mortar	mortar	production
				production		(RM)
				(RM)		
	0.000/	N/A	12 kg	0.00	_	
ABA	kg	11/21	12 Kg	0.00		
GGBES	0.180/	N/A	12 kg	2 16	_	
GODL2	kg	11/11	12 Kg	2.10	-	
OPC	3.000/	NI/A	272 kg	916.00	300 kg	900.00
	kg	11/7	272 Kg	810.00	500 Kg	900.00
Sand	0.0350/	N/A	900 kg	31 50	900 kg	31 51
Saliu	kg	11/21	900 Kg	01100	700 Kg	51.51
	$2.07/m^3$					
Water	<sup>-</sup> first	N/A	0.18 m <sup>3</sup>	0.37	0.18	0.37
	35m <sup>3</sup>					
Electricity						
Electric	0.380/	1	24 hours		_	_
oven	kWh	1	24 110013			
Electric	-first			9.69		
mixer	200kWh	0.5	3 hours		-	-
Total cost			859.72		931.88	

Table 6.3: Total Costing per 1000 kg of M-ASA GGBFS Mortars Production

# **CHAPTER 7**

#### **CONCLUSION AND RECOMMENDATIONS**

# 7.1 Conclusion

The results obtained from this study allow an evaluation of the performance of rendering mortars made of ASA and GGBFS. A lower mechanical strength of ASA mortars when compared to standard portland cement mortars is observed. However, high mechanical strength is not a strictly required property in mortar since mortar does not pose structural functions. In order to obtain sustainable construction materials, it is important to obtain materials with enhanced durability in order to assure a longer service life and to reduce maintenance costs. Water represents one of the main degradation factors for rendering mortars, which can seriously affect the material properties and reducing its durability.

Throughout this study, it is proven that ASA is able to be used as a partial replacement of cement in the production of mortar. The optimum performance of mortar incorporated with ASA and GGBFS is M-2ASA 4GGBFS with 19.67 MPa well above 12 MPa minimum strength required by the standard specification BS EN 1996-1-1: 2005, with workability of 156 mm, which is above the 120 mm  $\pm$  10 mm minimum workability requirement by the standard specification BS EN 1996-1-1: 2005. The setting time of cement mortar is also within the acceptable range. Durability of the cement is greatly increased as shown by the low water absorption coefficient and low porosity.

LCA was carried out to determine the environmental impacts of ASA cement mortar production from the extraction of raw materials for manufacturing to the end user. ASA cement mortar not only reduces carbon dioxide emissions from the calcination of limestone but also from burning fuel and by saving grinding energy in the clinker grinder. Therefore, the use of portland composite cement can be considered as more sustainable in comparison to OPC.

As a conclusion, incorporation of ASA as a partial replacement of cement in cement mortar production is able to reduce the volume of AS in landfill and to reduce the dependency of raw resources for cement production. Throughout this project, all of the objectives are achieved but further improvements are still required to determine the long-term effects of the ASA on the properties of mortar. Research on optimising the content of ASA in mortar and its long-term durability is also required.

#### 7.2 Recommendations

The study of utilisation of alum sludge as partial replacement of cement in mortar production is an innovative and sustainable product among construction and building materials. In order to discover and improve this research work in future, there are few aspects to be suggested for further improvement.

- Increase the percentage of alum sludge ash as partial cement replacement material to further study the development of mechanical properties and durability properties.
- Include mixing of admixture to reduce its setting time and increase it compressive strength.
- 3. Conduct a detailed study on leaching test and performance under fire to ensure product is able to attain serviceability and ultimate limit state.
- Study the possibility of alum sludge ash as cement admixture where percentage below 1 % is recommended in order to enhance the mechanical properties, when a reduced water repellent effect is acceptable.

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