School of Civil and Mechanical Engineering

Properties of Ambient Cured Alkali Activated Composites Using Waste Glass as a Partial Precursor and Fine Aggregate

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Dedicated

to my parents and brother for their tremendous love and encouragement

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to my lovely wife for her patience and sacrifices

Declaration

To the best of my knowledge and belief, this thesis contains no material previously published by any other person except where due acknowledgment has been made.

This thesis contains no material which has been accepted for the award of any other degree or diploma in any university.

Signature:

(MD NABI NEWAZ KHAN) 13/10/2021

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Abstract

The construction industry uses a significant amount of ordinary Portland cement (OPC) and natural aggregate every year due to the rapid growth of commercial and residential buildings. This led to increase of global carbon dioxide emissions (CO_2) and energy consumption, as well as decrease of natural resources. In this regard, the research on alternatives to conventional OPC and natural aggregate in construction sector have drawn keen interest of the Civil Engineering professionals and researchers during the past few decades. Recent research progress indicates that alkali activated composites (AAC) have a huge potential to complement the use of OPC in the construction sector. Generally, AAC is produced by a chemical reaction between the aluminosilicate based materials and alkaline solutions. Class F fly ash (FA) and ground granulated blast furnace slag (GGBFS) are the most commonly used aluminosilicate materials for making AAC. Meanwhile, a substantial amount of waste glass has been sent for landfilling which is not environmentally safe. Some previous studies have emphasized that waste glass can be used effectively as a partial precursor material or fine aggregate in AAC considering its physical and chemical characteristics. However, the use of waste glass powder (GP) as a partial precursor in AAC cured at ambient temperature needs a comprehensive investigation. Besides, the use of waste glass cullet as fine aggregate may cause alkali silica reaction (ASR) due to its reactive nature. Therefore, the present research aimed to evaluate the efficacy of waste glass as a partial precursor material and fine aggregate on the properties of AAC cured at ambient temperature. A series of experimental works were performed to study the viability of waste glass as a partial precursor material and fine aggregate in alkali activated systems.

The materials used in this study were Class F FA, GGBFS, OPC, GP, sodium hydroxide solution, commercial sodium silicate solution, silica sand, waste glass cullet and crushed granite aggregate. After receiving, the waste glass was ground in the laboratory using ball mill to obtain GP, whereas the unground waste glass cullet was used as fine aggregate. The physical, chemical and microstructural properties of the materials were evaluated at the first stage of experiment. In the second stage of the experiment, FA was replaced by 0% to 40% using GP to produce alkali activated FA and FA-GGBFS mortars cured at ambient conditions. The experimental results showed that the use of GP reduced workability of both FA and FA-GGBFS mortars. While no strength increase was observed by the use of GP in the FA mortars, 5% to 11% increase of compressive strength was noted in the FA-GGBFS mortars by the use

of 10% to 20% GP. The drying shrinkage of both FA and FA-GGBFS mortar increased with the increase of GP content. However, it was found that the inclusion of 10% to 20% GP reduced the porosity, sorptivity, chloride permeability and acid attack of FA-GGBFS mortars significantly. Microstructural investigations including the scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX), thermogravimetric analysis (TGA) and X-ray diffraction analysis (XRD) confirmed that the additional Si, Na and Ca elements provided by GP enhanced the formation of hydration products that enhanced the hardened properties.

The third phase of the experimentation focused on the effect of waste glass cullet as fine aggregate on the properties of FA and GGBFS blended mortars. Workability of mortar was found to increase by glass aggregate due to its smooth surface texture and low liquid demand. Compressive strength decreased from 77 MPa to 73 MPa at 90 days of age for replacing 100% sand by glass aggregate. Though porosity increased by 1.5% to 13% for using 25% to 100% glass aggregate, reductions were observed in drying shrinkage, sorptivity, chloride permeability and acid attack. This was attributed to the higher angularity and lower water absorption capacity of glass aggregate than natural sand. Besides, it was found that the use of 75% or 100% glass aggregate significantly improved the resistance against high temperature exposures. Microstructural studies revealed that mortars using glass aggregate had less micro cracks and enhanced bond at the interfacial transition zone (ITZ) after exposures to elevated temperature.

In the final stage of the experimental work the potential alkali silica reaction (ASR) of waste glass fine aggregate in alkali activated systems was evaluated by accelerated mortar bar test (AMBT) and concrete prism test (CPT). According to the test results, the expansions of waste glass fine aggregate exceeded the allowable limit of test standards when used in OPC and alkali activated GGBFS systems. However, the expansions of waste glass fine aggregate were within the safe limit when used in alkali activated FA or alkali activated FA-GGBFS blended systems. The alkali activated sample made of using 60% FA and 40% GGBFS in the binder and waste glass fine aggregate content of 100% exhibited the lowest expansion in both AMBT and CPT. The microstructural examinations revealed severe deterioration of glass aggregates when used in both OPC and alkali activated GGBFS systems due to the formation of calcium (Ca) rich expansive ASR gel. However, the gels formed at the aggregate paste interface of alkali activated FA and FA-GGBFS systems were not typical ASR gels, since they contained high Al/Si ratios and low Ca/Si ratios.

Overall, the outcomes of this study show that the use of waste glass as a partial precursor and fine aggregate in alkali activated systems is a promising option which can reduce the negative environmental issues and conserve natural resources.

List of Publications

The following publications have resulted from the work carried out for this degree.

<u>Journal:</u>

- M. N. N. Khan, J. C. Kuri and P. K. Sarker. Effect of waste glass powder as a partial precursor in ambient cured alkali activated fly ash and fly ash-GGBFS mortars. Journal of Building Engineering, 34 (2020) 101934.
- M. N. N. Khan and P. K. Sarker. Effect of waste glass fine aggregate on the strength, durability and high temperature resistance of alkali-activated fly ash and GGBFS blended mortar. Construction and Building Materials, 263(2020) 120177.
- M. N. N. Khan, A. K. Saha and P. K. Sarker. Evaluation of the ASR of waste glass fine aggregate in alkali activated concrete by concrete prism tests. Construction and Building Materials, 266 (2021) 121121.
- 4. **M. N. N. Khan** and P. K. Sarker. Alkali silica reaction of waste glass aggregate in alkali activated fly ash and GGBFS mortars. Materials and Structures, 52, 93 (2019).
- M. N. N. Khan, A. K. Saha and P. K. Sarker. Reuse of waste glass as a supplementary binder and aggregate for sustainable cement-based construction materials: A review. Journal of Building Engineering, 28 (2020) 101052.
- M. N.N. Khan, M. M. A. Elahi, J. C. Kuri, P. K. Sarker and F. U. A. Shaikh. Acid Resistance of Alkali Activated Composites Containing Waste Glass Fine Aggregate. Advances in Cement Research (Under Review).
- M. N. N. Khan, J. C. Kuri and P. K. Sarker. Sustainable use of waste glass in alkali activated materials against H₂SO₄ and HCl acid attacks. Cleaner Engineering and Technology (Under Review).

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LIST OF ABBREVIATIONS

AAC	Alkali Activated Composites
AMBT	Accelerated Mortar Bar Test
AS	Australian Standards
ASR	Alkali Silica Reaction
ASTM	American Society for Testing and Materials
BSD	Back-Scattered Electron Detector
C-(N)-A-S-H	Calcium-(Sodium) Aluminosilicate Hydrate
CAC	Calcium Aluminate Cement
C-A-S-H	Calcium Aluminium Silicate Hydrate
СН	Calcium Hydroxide
CPT	Concrete Prism Test
CRT	Cathode Ray Tube
C-S-H	Calcium Silicate Hydrate
EDX	Energy-Dispersive X-Ray
FA	Fly Ash
GGBFS	Ground Granulated Blast Furnace Slag
GP	Waste Glass Powder
ITZ	Interfacial Transition Zone
LOI	Loss on Ignition
МК	Metakaolin
M-S-H	Magnesium Silicate Hydrate
N-A-S-H	Sodium Aluminosilicate Hydrate
OPC	Ordinary Portland Cement
PDF	Powder Diffraction File
PVC	Polyvinyl Chloride

QXRD	Quantitative X-Ray Diffraction		
RCPT	Rapid Chloride Permeability Test		
RHA	Rice Husk Ash		
SCMs	Supplementary Cementing Materials		
SEM	Scanning Electron Microscope		
SF	Silica Fume		
SH	Sodium Hydroxides		
SS	Sodium Silicate		
SSD	Saturated Surface Dry		
TGA	Thermogravimetric Analysis		
VPV	Volume of Permeable Voids		
XRD	X-Ray Diffraction		
XRF	X-ray Fluorescence		

CHAPTER 1

INTRODUCTION

1.1 Background

Globally, construction industry consumes a vast amount of concrete every year, where ordinary Portland cement (OPC) is the major component of concrete. However, OPC manufacturing technique requires high energy and it emits a substantial amount greenhouse gases into the environment. Usually, the production of one tonne of OPC contributes about one tonne of carbon dioxide (CO₂) into the environment [1-2]. Therefore, research on the development of alternative binders to OPC has attracted special attention during the last few decades. The development of alkali activated composites (AAC) was first introduced by Purdon in the 1940s which has potential to reduce the negative effects related to the production of OPC [3]. Later, Glukhovsky and Davidovits have done extensive works towards the progress of AAC [3]. Generally, AAC is fabricated from the chemical reaction between the aluminosilicate-based industrial by-product materials and the commercial alkaline activators. These aluminosilicate-based materials are consisted of high SiO₂ compound and high or low in Al₂O₃ or CaO content, whereas commercially available aqueous solution of alkali hydroxide, silicate and carbonate are used as activators to activate the aluminosilicate-based materials [4-5].

Fly ash (FA) and ground granulated blast furnace slag (GGBFS) are the most widely used as the sources of aluminosilicate for the production of AAC [6-8]. It has been shown that FA and GGBFS based AAC has comparable mechanical properties and durability with those of conventional cement binders [9-11]. Generally, GGBFS is obtained as a by-product from the metal industries, whilst FA is received as a by-product from the coal-fired power stations. However, the generation of typical FA has been decreased noticeably in the recent times due to the decommissioning of coal-fired power stations globally. Although the current global production of GGBFS is about 300 million tonnes yearly, the availability of GGBFS might be limited in future due to its high demand as a construction material [12]. Furthermore, FA and GGBFS are not readily available in many parts of the world. Consequently, numerous research attempts have been carried out using other waste or by-products such as silica fume (SF), rice husk ash (RHA) and palm oil fuel ash (POFA) as partial precursor materials in preparation of AAC [13-15]. The outcomes of the previous research attempts show that a material which have abundance of reactive silica may be used as a partial precursor of AAC. In this connection, waste glass powder (GP) has a potential to be used as a precursor material in AAC since it contains a significant amount of silica in the amorphous phase [16]. Generally, GP is produced by grinding rejected or non-recyclable glass items. Globally, more than 14 million tonnes of waste glass is being sent for landfilling every year, which poses a severe threat to the environment as shown in Figure 1.1 [17-19]. However, the total recycling rate of waste glass is not even satisfactory in the developed countries such as USA, UK and Australia as displayed in Table 1.1 [18]. For example, only 42% of total waste glass is recycled in Australia. Therefore, recently, the reuse of GP as a partial precursor material in AAC is considered as an effective way to raise the use of waste glass [20-21]. Some previous works explored that the inclusion of certain dosages of GP as a partial substitution of traditional precursor materials provide several advantageous on the properties of AAC due to its tendency to dissolute in alkaline medium [22-23].



Fig. 1.1 Landfilling using waste glass [19]

Countries	Waste glass generation	Landfilling by waste	Recycling rate
	per year (tons)	glass (tons)	(%)
Iran	740,000	666,000	5
Hong Kong	109,500	98,550	10
China	40,000,000	5,200,000	13
Egypt	3,450,000	2,898,000	16
USA	20,000,000	5,400,000	27
Singapore	72,800	51,688	29
UK	770,000	531,300	31
Australia	800,000	336,000	42
India	21,000,000	11,550,000	45
Turkey	120,000	39,600	67
Canada	116,000	37,120	68
Netherlands	20,000	2000	90

Table 1.1 The amounts waste glass produced and recycled in various countries [18]

Meanwhile, natural sand has been considered as the most suitable and popular option for fine aggregate in the construction industry due to its inert chemistry and high availability. Since natural sand is the primary source of fine aggregate for construction purposes, excessive sand mining occurs in many countries to meet the high demand of sand. This has harmful effects to the aquatic life and equilibrium of hydraulic structures [24]. Consequently, the high consumptions of natural fine aggregate have significant impacts on the sustainability of construction practices. In this connection, research on alternative materials to natural fine aggregate has also received the highest attention during the past few decades [25-26]. Consequently, some studies investigated the suitability of various by-products or waste materials such as waste glass cullet [27], recycled plastic [28], and ferronickel slag [29] as a partial or total replacement of natural fine aggregate in the production of concrete. Considering the availability in large volumes as well as the physical characteristics, waste glass cullet has a potential to be used as a fine aggregate as reported in some recent research works [30-31]. Therefore, this research work focused to uncover the efficacy of waste glass as a partial binder and fine aggregate in alkali activated binder based concrete.

1.2 Research objectives

The principal aim of this thesis is to evaluate the efficacy of waste glass as a partial precursor and fine aggregate materials for making AAC. The specific objectives of this thesis are as follows:

- Conduct a comprehensive literature review to understand recent research progress on reuse of waste glass as a construction material.
- Evaluate the influence of GP as a partial precursor on the workability, compressive strength and microstructures development of AAC.
- Investigate the durability performance such as drying shrinkage, sorptivity, porosity, chloride permeability and acid resistance of AAC containing GP.
- Evaluate the impact of waste glass cullet as fine aggregate on the workability, compressive strength and microstructure of AAC.
- Examine the influence of waste glass fine aggregate on the durability performance such as drying shrinkage, sorptivity, porosity, chloride permeability and acid resistance of AAC.
- Evaluate the potential alkali silica reaction (ASR) of waste glass fine aggregate in AAC by accelerated mortar bar test (AMBT) and concrete prism test (CPT) methods.

1.3 Research significance

The safe disposal or value-added utilisation of waste and by-product materials has been a major topic of interest among researchers and industry owners during the past few decades since the on-site or off-site dumping of these waste materials is not only hazardous to the environment but also land, labour and cost intensive. About 46 millions metric tons of waste glass is generated worldwide every year, out of which only about 10% is recycled or reused in the manufacturing of new glass products and the remaining greater portion is mainly sent to landfill sites [32]. However, the landfilling using waste glass is considered as a severe threat to the environmental equilibrium since glass substances are commonly non-biodegradable in nature and can cause leaching of heavy metals. Thus, the use of waste glass as a construction material can be considered as an effective approach to reduce the risk of environmental pollution and serve as a feasible alternative to the use of natural resources. Some previous studies showed that waste glass can be used as a partial replacement of common precursor materials such as FA and GGBFS for making AAC due to the presence of high silica, sodium and calcium contents in waste glass. However, the number of conducted research studies on the feasibility

of using waste glass in AAC is very limited. Besides, several important parameters or properties were not covered or investigated adequately in the previous research works. Consequently, a critical discussion on the relevant previous works is presented in Chapter 2 in order to identify the research gaps and to highlight the importance of the present research. Furthermore, it is essential to conduct a comprehensive investigation before using any by-product or waste material as a component of AAC since the properties of the waste materials can vary significantly depending on the source and processing. Therefore, this study aimed to evaluate the fresh and mechanical properties and durability of AAC using waste glass as a partial precursor and fine aggregate.

1.4 Thesis outline

This thesis is prepared by following the thesis guidelines of the Curtin University, Western Australia. The findings of series of experimental work and related discussion are presented from Chapter 3 to Chapter 8. The detail of this thesis structure is described as followings:

Chapter 1 covers background, objectives and significance of this study.

Chapter 2 presents a comprehensive literature review related to the reuse of waste glass as partial binder and fine aggregate materials in both OPC system and AAC.

Chapter 3 presents the experimental studies to evaluate the efficacy of GP as a partial precursor on the workability, compressive strength and durability of ambient cured alkali activated FA and GGBFS blended mortars.

Chapter 4 focuses on the influence of GP on the acid resistance of ambient cured alkali activated FA and GGBFS blended mortars.

Chapter 5 describes the experimental works to assess the influence of waste glass cullet as fine aggregate on the workability, compressive strength and durability performance of ambient cured alkali activated FA and GGBFS blended mortars.

Chapter 6 investigates the effects of waste glass cullet as fine aggregate on the acid resistance of ambient cured alkali activated FA and GGBFS blended mortars.

Chapter 7 presents the experimental program to evaluate the potential alkali silica reaction (ASR) of waste glass fine aggregate in alkali activated systems by AMBT.

Chapter 8 examines the potential ASR of waste glass fine aggregate in alkali activated systems by CPT.

Finally, **Chapter 9** summarizes the significant findings of this research and provides some recommendations for future work.

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CHAPTER 2

LITERATURE REVIEW

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This chapter presents a literature review on the reuse of waste glass as a supplementary binder and aggregate in both cement based and alkali activated systems. The outcomes of the previous studies are critically analysed, discussed and gaps identified.

2.1 Overview

It is well known that the utilization of various by-product materials such FA, GGBFS, RHA, SF and POFA in concrete provides environmental, economic and engineering benefits [1-5]. Thus, research on the use of various waste materials and their application as construction materials in different ways have good prospects of bringing environmental, technical and economic benefits. Although research on waste glass as a construction material started in 1960s [6-9], the current research trend indicates that waste glass may be considered as a suitable construction material because of its flexible size and shape, chemical compositions and widespread availability when compared with other supplementary cementing materials (SCMs) [10-12]. On the other hand, hundreds of thousands tonnes of glass are being stockpiled and landfilled globally every year which is a serious threat to the environment. Since glass is a nonbiodegradable material, it takes one million years to break down naturally [13]. Therefore, nowadays, the recycling of waste glass has become a major concern to the scientific community. In this connection, a significant number of research works studied the use of waste glass in cementitious systems. It has been shown in literature that waste glass was used in ordinary Portland cement (OPC) based mortar and concrete in six possible ways, such as (i) raw material for cement production [14-16], (ii) partial replacement of cement [17-19], (iii) partial or full replacement of fine aggregate [20-22], (iv) combined partial replacements of cement and fine aggregate [23-25], (v) partial or full replacement of coarse aggregate [26-28] and (vi) combined partial or full replacements of coarse and fine aggregates [29-31]. It has been reported that the use of crushed waste glass as coarse aggregate and ground waste glass as a raw material in cement production are less attractive due to (i) the high risk of ASR induced damage and substantial loss to the mechanical properties when waste glass is used as coarse aggregate and (ii) high alkali content (Na₂O) of ground glass may contaminate the clinker if it

is used as a raw material for cement production [32]. Recently, it has been noticed that some researchers attempted to use waste glass in preparation of AAC in different ways such as (i) a sole precursor material to produce alternative sodium silicate solution [33], (ii) a partial replacement of traditional precursor materials such as FA and GGBFS [34], and (iii) fine aggregate [35]. However, the synthesis of alternative sodium silicate solution using GP is a complex and energy intensive process since it requires thermal treatment and multiple mechanochemical steps [36]. Moreover, the number of studies related to the utilization of waste glass as a supplementary binder material and fine aggregate in the production of OPC based composites or AAC have been increasing noticeably due to its huge potential. Therefore, the main motivation of this chapter is to review the previous studies related to the fresh, mechanical properties and durability of OPC based and alkali activated based mortar and concrete containing waste glass as supplementary binder and fine aggregate to establish the gaps of knowledge in the relevant area.

2.2 Use of GP as a SCM in OPC based mortar and concrete

2.2.1 Factors affecting the mechanical properties

A significant number of studies used waste GP as a SCM in preparation of concrete and mortar [37-39]. It can be observed that the use of ground waste glass as a partial replacement of cement in concrete and mortar influenced the mechanical properties significantly by the pozzolanic reaction of fine glass particles. This pozzolanic reaction of GP depends on several factors such as the content, glass colour and type, particle size, curing age curing temperature. The influences of these factors on the properties of glass blended mortar or concrete mixes are discussed in the following subsections.

2.2.1.1 Effects of type and colour of glass

Some researchers studied the effect of glass colour and glass type on the strength development of glass blended cementitious mixtures. Dyer and Dhir [37] found that mortar with 10% white and green GP showed about 27% and 20% increment in compressive strength at 28 days as compared to the control mortar. However, the mortar with 10% amber GP achieved only about 2% higher compressive strength than the control mortar as shown in Fig. 2.1. Pereira-de-Oliveira et al. [38] also reported the similar phenomena. Karamberi and Moutsatsou [39] observed that compressive strength of mortar containing 25% flint GP was about 34.5% higher than the mortar containing 25% amber glass. The authors [39] predicted that the chemical reaction of the basic coloration elements of glass particles with calcium hydroxide (Ca(OH)₂)

might affect the strength development. Although amber GP showed higher reactivity than the flint or green GP in pozzolanic activity tests as shown in Fig. 2.2, the authors revealed that amber glass particles were less active from the microstructure studies of cement paste containing amber GP.



Fig. 2.1 28-day compressive strength of GP blended mortar [37]



Fig. 2.2 Results of the pozzolanic activity rate test [39]

Therefore, the mortar containing amber GP showed lower compressive strength than the mortar with flint GP at similar age and cement replacement level. Similarly, Mirzahosseini and Riding [40] reported that green GP mixed mortar exhibited about 3% higher compressive strengths than the clear GP mixed mortar at 91 days. The authors stated that the silicon and aluminium dissolution of green GP is high, which results in increased pozzolanic reaction under high pH of the cementitious matrix [40]. Furthermore, Bignozzi et al. [41] explained that glass dissolution rate increases with the increase of glass modifiers PbO + Na₂O_{equivalent} and with the decrease of both glass formers, $SiO_2 + Al_2O_3$ and glass stabilizers, CaO + MgO contents. Therefore, GP associated with higher amount of glass modifiers and lower amount of glass formers and glass stabilizers accelerate alkali silica reaction instead of promoting pozzolanic reaction by increasing the amount of dissolved K, Na, Pb and Si atoms. As a result, the authors found that soda-lime GP mortars showed about 4% higher 90-day compressive strength than the crystal GP mortars, where soda-lime GP consisted of lower amount of glass modifiers (13.91%) and higher amount of glass formers (72.46%) as compared to crystal GP. Besides, AL-Zubaid et al. [42] reported that neon GP blended concrete showed about 9.5% greater compressive strength than the concrete with green GP at 13% cement replacement level due to the presence of higher amount of SiO₂ (68.2%) and CaO (22.6%) in neon GP. Recently, Lee et al. [43] reported that concrete containing waste glass sludge displayed about 10% higher compressive strength than concrete containing waste GP at 20% cement replacement level due to greater pozzolanic reactivity and finer granulometry of waste glass sludge. On the other hand, Islam et al. [44] stated that the difference in quantities of individual chemical compounds between clear and coloured GP is minor. Therefore, the authors studied the influence of GP on the workability and compressive strength of mortar and concrete using clear GP only. It can be seen from majority of the studies that the type and colour of glass could have significant influence on the strength development of blended mortar and concrete due to the variation of chemical composition or the nature of reaction of different colour and type of GP.

2.2.1.2 Effect of particle size

Particle size of the GP plays a vital role on the strength development of glass blended mortar and concrete. Shao et al. [45] found that the concrete containing 30% cement replacement by glass particles of 38 μ m size showed 14.3% and 33.3% higher compressive strengths than the concretes containing glass particles of 75 μ m and 150 μ m, respectively, as presented in Fig. 2.3.



Fig. 2.3 Effect of the particle size of GP as SCM on compressive strength

Khmiri et al. [46] studied compressive strengths of concrete containing ground glass of four different particle sizes, namely 80-100 μ m, 40-80 μ m, <40 μ m and <20 μ m. The authors found that the strength of GP blended concrete increased with the decrease of particle size as depicted in Fig. 2.3. The authors also reported that concrete with 20% GP with particle size of 20 μ m showed about 4% higher compressive strength than the control mortar at 90 days. Similarly, Mirzahosseini and Riding [47] examined the influence of glass particle size on compressive strength of mortar using particle size ranges of 63-75 μ m, 25-38 μ m and 0-25 μ m. The authors reported that the mortar with GP of particle sizes less than 25 μ m showed about 30% higher compressive strength than the mortar containing GP of particle sizes 63-75 μ m at similar cement replacement level and curing ages. Recently, Lu et al. [48] used ground glass with particle sizes of 28.3 μ m, 47.9 μ m, 88.5 μ m and 204 μ m. The authors found that the mortar with GP of 28.3 μ m showed about 40% and 35.8% higher compressive and flexural strengths than the mortar with GP of 204 μ m size. In addition, Parghi and Alam [49] found that the mortar with 30% cement replacement by GP of 75 μ m size showed 37.5% higher compressive strength and 41.7% higher flexural strength than the control mortar at 90 days. Some

researchers used very fine GP (average particle sizes $<20 \ \mu$ m) as SCM in concrete and mortar. The authors found that compressive, flexural and tensile strengths of concrete and mortar increased significantly due to higher pozzolanic reaction and filler effect of the finer glass particles [50-55]. However, Matos and Sousa-Coutinho [56] found about 3.5% lower compressive strength and 6.25% higher flexural strength of mortar for 20% cement replacement by GP than the control mortar, when the average particle size of glass was very close to that of cement. Similarly, Nassar and Soroushian [57] found that concrete containing GP of median particle size 13 µm showed about 3% lower compressive strength and 5% lower flexural strength as compared to the control concrete. This may be attributed to the use of 50% recycled coarse aggregate in the GP blended concrete. From the above discussion, it can be concluded that compressive, flexural and tensile strengths of concrete and mortar increased with the decrease of particle size of glass and it was more pronounced when the particle size was in the range of 20 to 45 µm.

2.2.1.3 Effect of curing age

Curing age can significantly affect the strength gain of blended cement concrete. Shao et al. [45] reported that compressive strength of GP mixed concrete increased with the increase of age and gained higher strength than the control concrete at 90 days. Similar trend was observed for GP mixed concrete by Pavoine et al. [58] and GP blended self-compacting mortar by Sharifi et al. [59]. On the other hand, Schwarz et al. [60] found lower compressive strength for GP blended concrete compared to normal concrete at various ages up to 90 days. Some studies reported that GP mixed mortars showed lower compressive strength at early ages than the control mortar; however, exhibited higher compressive strength at 28 days as compared to the control mortar [61-62]. Usually, cementitious mixtures achieved strength mainly from the hydration of cement, whereas GP blended mixtures gained strength as a result of both hydration and pozzolanic reactions. Pozzolanic activity of GP is low at the early ages, therefore, glass blended mixtures showed lower strength at the early ages [62]. In addition, Idir et al. [63] found that 30% GP blended mortars showed lower compressive strength than the control mortar until 90 days of curing; however, displayed about 4% higher compressive strengths than that of the control mortar at 210 days. Calmon et al. [64] and Harbi et al. [65] also noticed similar phenomena for GP mixed mortar and concrete. This is attributed to the more pronounced pozzolanic activity of GP at the later ages. The study by Korjakins et al. [51] reported that compressive strength of GP mixed concrete gradually increased up to 365 days of curing and showed higher compressive than the control concrete at all curing ages in between 7 to 365
days. Besides, some researchers noticed that GP blended mixes showed significantly higher compressive strength than the control mix only at 365 days [44, 54, 66]. For instance, Du and Tan (2017) found that concrete containing 15-60% GP showed about 27-12% higher compressive strength than the control concrete at 365 days, as shown in Fig. 2.4 [54].



Fig. 2.4 Compressive strength development of GP mixed concrete with age [54]

The authors explained that the reactive silica present in GP slowly dissolved with prolonged curing under the alkaline environment and reacted with Ca²⁺ to form calcium silicate hydrate (C-S-H) gel, which increased strength by the reduction of porosity [54]. In addition, Matos and Sousa-Coutinho [56] found that mortar samples with 10% GP gained significant compressive and flexural strengths up to 562 days; however, strengths were about 5% lower than the strength of control mortar. Furthermore, Carsana et al. [67] noticed no increase or decrease of compressive strength even after 7 years of age in mortar specimens containing GP. Therefore, in most cases, GP blended mixtures exhibited good mechanical properties at the later ages due

to pozzolanic reaction of the fine glass particles and it exceeded the strengths of control mixtures.

2.2.1.4 Effect of curing temperature

Some researchers studied the influence of curing temperature on the strength development of GP blended mixtures [40, 66, 68-69]. Mirzahosseini and Riding [40] reported that mortars with 25% GP cured at 50°C displayed about 25% higher 91-day compressive strengths than that cured at 23°C. The authors found that paste sample with 25% GP showed about 19% lower calcium hydroxide (CH) content than the control paste sample cured at 23°C after 91 days, whereas the paste sample with 25% GP exhibited about 34% lower CH than the control paste cured at 23°C for 91 days. Therefore, GP blended samples cured at higher temperature achieved higher compressive strength due to the improved pozzolanic reaction of glass. Besides, Kou and Xing [66] investigated compressive and flexural strengths concrete containing 15% and 30% GP at various curing ages between 1 day and 365 days, when specimens were cured at 20°C and 90°C for the first 24 hours. It was found that the GP mixed concrete specimens cured at 90°C showed higher compressive and flexural strengths at all ages than the specimens cured at 20°C. Furthermore, Shi et al. [68] investigated compressive strength of GP mixed mortars at temperatures of 23°C, 35°C and 65°C. The authors reported that the GP mixed mortars achieved higher compressive strength than the control mortar even at early ages (<7 days) cured at 35°C and 65°C, as presented in Fig. 2.5. The authors stated that heat curing conditions provided higher apparent activation energy for the pozzolanic reactions between GP and lime than that for the hydration of Portland cement. Besides, Liu et al. [69] concluded that higher curing temperatures can promote both the hydration of cement and the pozzolanic reaction of GP at the same time, as a result, GP blended mixes showed higher compressive strength at higher curing temperatures.

Generally, the strengths of the mixtures containing GP were satisfactory at ambient curing condition; however, higher strengths were observed for higher curing temperatures due to the accelerated pozzolanic activity of glass particles.





2.2.1.5 Effect of GP content

Previous studies show a debate among researchers about the optimum percentage of cement replacement by GP. Jang et al. [70] used 3%, 5% and 10% GP as a SCM in preparation of mortars. The authors found that GP blended mortars showed higher compressive strengths up to 10% cement replacement level as compared to the control mortar. The authors also revealed that mortars containing GP showed relatively dense microstructure with fewer and smaller pores because of the formation of secondary C-S-H gel by pozzolanic reaction. Some researchers found that GP mixed concrete and mortar showed 10-20% higher compressive, 15% higher flexural and 10-15% higher tensile strengths than the control concrete for up to 10% cement replacement level [59, 71]. In addition, Kamali and Ghahremaninezhad [72] found that 20% GP mixed specimens showed 24% higher 91-day compressive and flexural strengths than the control specimens. Similar observations were also reported by Aliabdo et al. [73]. The authors added that strength development might be affected negatively if the GP content is more than 20% due to agglomeration of glass particles. However, Calmon et al. [64] found that GP

blended mortars showed about 6% higher compressive strength than the control mortar only at 270 days and 5 % cement replacement level. The authors claimed that higher concentration of Na₂O (15.41%) and Al₂O₃ (6.07%) present in windshield waste glass produced some delayed ettringite. Therefore, the GP blended mortars showed lower compressive strength than the control mortars at 10% to 20% of cement replacement levels. Some researchers used high volume GP in preparation concrete and mortar [50, 74-75]. Among these studies, Zhi et al. [50] noticed no significant change of compressive strength even at 25% cement replacement level as compared to control specimen, whereas Ozkan and Yuksel [74] and Wang [75] found that GP blended specimens showed negligible increase of strength (<5%) only at 10% level of cement replacement. In contrast, Du and Tan [54-55] found that GP blended concrete exceeded the compressive strength of control concrete by 12% after 1 year at 60% cement replacement level. The authors stated that high volume GP (≤60%) provided more compact microstructure at the interfacial transition zone (ITZ) of the paste matrix. As a result, compressive strength increased comprehensively. On the other hand, Cassar and Camilleri [76] found that concrete containing 10-50% GP showed lower compressive and flexural strengths than the control concrete due to poor pozzolanic activity by coarser sizes of glass particles (particle size ranges 100-600 µm). Therefore, it can be concluded that strengths of concrete and mortar significantly increased with the increment of the GP content. In most cases, this increment trend is pronounced up to 30% cement replacement level if finer sizes (particle sizes of <45 µm) GP was used.

2.2.2 Durability properties

2.2.2.1 Water absorption

The effects of using GP as SCM on the water absorption of concrete and mortar are presented in Table 2.1. It has been reported that water absorption of concrete and mortar decreased significantly with the increment GP content due to the higher pozzolanic reaction [49, 57, 59, 73]. Parghi and Alam [49] found that the mortar containing 30% GP showed about 17.4% less water absorption than the control mortar at 28 days. Similarly, Nassar and Soroushian [57] reported that concrete containing 20% GP showed about 13.1% less water absorption as compared to the reference concrete at 56 days. The authors stated that the use of fine glass particles with a median size of 13 μ m as SCM improved the particle packing by filling effect which introduced a dense and less permeable microstructure.

Source of GP	Optimum results		Age	Water absorption	Df
	GP (%)	Particles size (µm)	(days)	Decrease (%)	Kelerences
Glass bottles	30	<75	28	17.4	[49]
Waste glass	20	13	56	13.1	[57]
Waste glass	20	<100	90	13.6	[59]
Glass containers	20	<75	56	17.7	[73]

Table 2.1 Effect of GP as SCM on the water absorption of mortar and concrete

Therefore, the GP blended concrete exhibited lower water absorption than the control concrete. Besides, Aliabdo et al. [73] found that the concrete with 20% GP exhibited about 17.7% less water absorption as compared to the control samples. In contrast, Sharifi et al. [59] reported that GP mixed concrete showed higher water absorption beyond 20% cement replacement level as compared to the control concrete due to the decrease of hydration product. Furthermore, Mirzahosseini and Riding [40, 47] reported that GP blended mortar showed significant reduction in water sorptivity at 91 days due to the denser microstructure as a result of higher amount of hydration products.

2.2.2.2 Drying shrinkage

The drying shrinkages of mortar and concrete containing GP as SCM are shown in Table 2.2. Wang [75] and Wang et al. [77] reported that the mortars with 20% GP of fineness $650 \text{ m}^2/\text{kg}$ showed about 6.9% less drying shrinkage as compared to the control mortar at 28 days. In addition, Sharifi et al. [59] and Sadati and Khayat [78] found that drying shrinkage of waste GP blended specimens decreased with the increase of age and the increase of glass content.

|--|

Course of CD	Optimum results		Age	Length	Defense	
Source of GP	GP (%)	Particles Size (µm)	(days)	Increase (%)	Decrease (%)	Kelerences
Waste glass	30	<100	91	-	23.75	[59]
Vehicle windshields	5	<75	63	6	-	[64]
TFT-LCD glass	20	-	28	-	6.9	[75, 77]
Glass fiber	40	-	91	-	19.6	[78]

TFT-CRT - Thin film transistor liquid crystal display

Sharifi et al. [59] found that the concrete containing 30% GP showed about 23.75% reduction in shrinkage and concrete containing 50% GP showed about 20% reduction in shrinkage at 91 days as compared to that control concrete. The authors stated that GP blended concrete achieved lower shrinkage due to the consumption of free water by fine glass particles to produce C-S-H during pozzolanic reaction [59]. Similarly, Sadati and Khayat [78] found that the mortar with 40% GP of fineness 600 m²/kg showed almost 20% reduction in drying shrinkage than the control mortar at 91 days. However, Calmon et al. [64] reported that mortars containing 5-20% GP with mean particle sizes of <75 μ m showed about 6-16% higher shrinkage than the control mortar at 63 days. Similar observations were reported by Omran &Tagnit-Hamou [52] and Omran et al. [53]. The authors explained the reason as the use of GP in concrete as a replacement of cement leads to excess water in the concrete due to low absorption of glass particles, which resulted in higher drying shrinkage.

2.2.2.3 Alkali silica reaction (ASR)

It is well known that the expansion induced by ASR is strongly influenced by reactive particles. It is also known that GP is rich in both alkali and amorphous silica. Therefore, it can promote either pozzolanic reaction or formation expansive product when used as an SCM in cementitious systems [41]. In this connection, a significant number of studies conducted ASR test of mortar as per accelerated mortar bar test (AMBT) when ground waste glass is used as an SCM and the summary of the reported results are presented in Table 2.3. It can be noticed from Table 2.3 that the use of GP as an SCM in mortar and concrete significantly reduced the expansion by ASR. The ASR of GP blended cementitious mixtures depends on several factors including fineness, content, colour and type of the used glass. Shao et al. [45] found that the mortar made of GP having particle sizes 38 µm showed lower expansions than the mortar containing GP having particle sizes of 75 µm and 150 µm. The authors also found that the addition of 30% GP of 38 µm size reduced more than 40% expansion as compared to the control mortar after 16 days in AMBT. Similar observations were reported by Pereira-de-Oliveira et al. [38], Nassar and Soroushian [57], Schwarz et al. [60] and Jang et al. [70] for 10-30% replacement of cement by GP. Besides, Dyer and Dhir [37] found that mortars with 40% GP with 21% particles less than 45 µm reduced the expansion by more than 90% as compared to the control mortar. The authors explained that the reduction of particle size of glass provides greater surface area for relatively rapid reaction in presence of the available calcium in solution. Some researchers also reported that expansion of glass blended mixtures decreased with the increment of glass content [50, 59, 61, 71]. Wang et al. [50] stated that highly reactive GP reacted with lime and formed C-S-H gel with a low C/S ratio. As a result, the available alkalis decreased for potential ASR. Furthermore, some studies addressed the influence of glass colour and type on the ASR of mortars containing GP [37-39, 68].

	Optimum results		Age	Expa		
Source of GP	GP	Particles	(days)	Increase	Decrease	- References
	(%)	size (µm)		(%)	(%)	
Coloured glass	40	21%<45	16	-	93.8	[37]
Green glass	30	45-75	14	-	37.8	[38]
Waste glass	25	10-20	14	6.25	-	[41]
Fluorescent glass	30	38	16	-	46.3	[45]
Glass bottles	5	75	14	-	25	[49]
Ground glass	50	-	21	-	81	[50]
Car windscreens	10	-	14	-	76.8	[56]
Powdered glass	20	13	28	-	40.9	[57]
Waste glass	25	<100	42	-	27.4	[59]
Window glass	20	25%<10	21	-	42.9	[60]
Waste glass sludge	20	-	16	-	73.5	[61]
Glass beads	20	-	21	-	52.7	[68]
LCD glass	10	88	35	-	42.9	[70]
Waste glass	20	<45	14	-	70.5	[71]
Glass bottles	30	-	14	-	4.2	[74]

Table 2.3 Effect of GP as SCM on the ASR of mortar and concrete

LCD - Liquid crystal display

Dyer and Dhir [37] reported that green GP mixed specimens showed almost seven times less expansions than that of white GP mixed specimens in ASR test at a similar cement replacement level and curing age. Similarly, Karamberi and Moutsatsou [39] concluded that mortar with amber GP performed well against ASR due to the hydrolysis of the large amount of Fe^{3+} in the network of the amber glass as compared to the Fe_{total}/Fe^{2+} ratio in the green and flint glasses. However, Pereira-de-Oliveira et al. [38] found no significant difference in mortar expansions with amber, green and flint glasses at the same cement replacement level. However, Bignozzi et al. [41] found that GP blended mortars exhibited higher expansion compared to the control mortar. According to their study, crystal glass mortar showed the highest expansion due to the high content of dissolved K, Na, Pb and Si. However, fluorescent glass mortar and soda lime glass mortar expanded less due to the high content of glass stabilizers (CaO + MgO), which reduced ASR by delaying the alkalis and silica dissolution.

2.2.2.4 Resistance to chloride attack

Table 2.4 presents the performance of GP blended mortar and concrete against chloride attack. It was generally found that the resistance to chloride attack was enhanced by significant pozzolanic activity of fine glass particles. The influence of waste glass as SCM in mortar and concrete against chloride attack mainly depends on glass content, fineness and age of specimens [50, 54-55, 79].

~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~	Optimum results		Age	Chloride permeability	
Source of GP	GP (%)	Particles size (µm)	(days)	Decrease (%)	- References
Ground glass	50	-	90	76.2	[50]
Soda-lime glass	60	-	56	92	[54-55]
Window glass	20	-	56	51.9	[56]
Waste glass	20	13	28	56	[57]
Ground glass	20	12.3	91	75.3	[58]
Window glass	10	25%<10	90	17.4	[60]
Recycled glass	20	8.4	90	80.9	[72]
Glass bottles	10	-	28	16.3	[74]
Coloured glass	10	100-600	28	46.7	[76]
Green glass bottles	30	-	28	57.5	[79]

Table 2.4 Effect of GP as SCM on the chloride resistance of mortar and concrete

Schwarz et al. [60] and Ozkan, and Yuksel [74] found that the specimens with 10% GP exhibited about 17% higher resistance to chloride ion penetration than the control specimen. Some researchers reported that GP mixed samples showed more than 50% higher resistance to chloride attack than the control sample at 20% cement replacement level [56-58, 72]. Besides, Wang et al. [50] and Du and Tan [54] found 76.2% and 92% greater resistance to chloride attack at 50% and 60% cement replacement by GP. In addition, Carsana et al. [79] reported that the mortar with GP with fineness of 600 m²/kg showed higher resistance against chloride attack compared to the mortar with GP of fineness 400 m²/kg at a similar cement replacement level. Furthermore, Nassar and Soroushian [57] concluded that the inclusion of fine GP having

a mean particle size of 13 μ m increased the resistance to chloride permeation in concrete by the pore refinement, pore blocking and filling effects of glass pozzolanic reaction product.

2.2.2.5 Resistance to sulphate attack

Only a few studies examined the performance of GP blended concrete and mortar against sulphate attack [65, 74, 79] and the summary of the reported results in these studies are provided in Table 2.5.

	Optimum results		Age	Sulphate			
Source of GP	GP	Fineness	(days)	Increase	Decrease	- References	
	(%)	(m ² /kg)		(%)	(%)		
Glass bottles	25	506.7	90	-	6.25	[65]	
Glass bottles	10	483	28	31	-	[74]	
Green glass bottles	30	600	60	88	-	[79]	

Table 2.5 Effect of GP as SCM on the sulphate resistance of mortar and concrete

Ozkan, and Yuksel [74] found that the mortar with 10% GP of fineness 483 m²/kg showed 31% higher compressive strength when exposed to sodium sulphate (Na₂SO₄) solution; however, it exhibited negligible increase in strength (<2%) when exposed to magnesium sulphate (MgSO₄) solution as compared to the control mortar. The authors explained that the additional C-S-H gel produced by pozzolanic reaction of GP increased the resistance to Na₂SO₄ attack but not to MgSO₄ attack since C-S-H gel is converted to non-cementitious magnesium silicate hydrate (M-S-H) gel in the presence of magnesium sulphate solution. Similarly, Carsana et al. [79] found that mortars with 30% green GP of fineness 600 m^2/kg showed 80% less expansion than the control mortar even after exposed to Na₂SO₄ solution for 60 days. In contrast, Harbi et al. [65] found that mortars containing 25% GP of fineness 507 m²/kg showed about 6.25% less tensile strength by bending tests as compared to the reference mortar after immersion in Na₂SO₄ solution for 90 days. According to their study [65], glass mortars showed good resistance against sulphate attack up to 90 days due to formation of gypsum from the reaction between Na₂SO₄ and Ca(OH)₂ which reduced the micropores and provided a denser structure. However, resistance against sulphate attack decreased at 180 days due to the formation of expansive sulphated hydrate, which introduced microcracks.

2.3 Use of waste glass cullet as fine aggregate in OPC based mortar and concrete

2.3.1 Factors affecting the mechanical properties

Numerous studies have been conducted using broken waste glass as a substitute for natural sand in mortar and concrete [80-82]. The mechanical properties of mortar and concrete containing glass fine aggregate depend on several factors such as the quantity, particles size, curing age, type and colour due to its possible reactivity. The effects of different factors related to glass fine aggregate on the mechanical properties of mortar and concrete are discussed in the following subsections.

2.3.1.1 Glass colour and glass type

Tan and Du [80] found that mortars containing 25-100% green glass fine aggregate showed about 20-25% higher compressive, 20-25% higher flexural strength and 7-16% higher tensile strength than the mortars with the same percentages of clear glass fine aggregate. The authors revealed based on microscopic analysis that the presence of micro-cracks in clear glass fine aggregates were more obvious than in the green glass fine aggregates [80]. Therefore, the mortars with green glass fine aggregate showed better mechanical properties than the mortars with clear glass fine aggregate. However, Park et al. [81] did not notice any significant influence of glass colour on the strength development of concrete containing glass fine aggregates of different colours. In another study, Chen et al. [82] reported that concrete with 40% electronic grade waste glass (E-glass) fine aggregate showed about 40% higher compressive strengths at 365 days as compared to control concrete. The authors stated that Eglass particles might have participated in both hydration and pozzolanic reaction due to the presence of relatively higher amount of SiO_2 (54%) and Al_2O_3 (15%) in waste E-glass, which has positively influenced the strength development. Similarly, Mageswari and Vidivelli [83] found that the use of sheet glass as fine aggregate improved compressive, flexural and tensile strengths of concrete. In contrast, Ling and Poon [84] found that concrete containing 25-100% treated (acid washed) funnel glass or non-treated (without acid treatment) funnel glass or beverage glass fine aggregate showed about 5-20% lower compressive strength and 5-30% lower flexural strength than the control concrete. The authors mentioned that the weaker adhesion between the smooth surface of glass particles and cement paste was mainly responsible for the lower strength values of glass fine aggregate based concrete [84]. Furthermore, Patel and Dalal [85] used a mixture of glass and polyvinyl chloride (PVC) as replacement of sand. The authors reported that compressive and flexural strengths decreased with increasing the mixture of glass and PVC content. From microstructural analysis, the authors observed that glass particles were distributed evenly; however, PVC particles formed clusters at some spots which might be responsible for the decrease of strengths.

2.3.1.2 Particle size

Corinaldesi et al. [86] used finely ground glass as micro fine aggregate with average particle sizes of 36, 36-50 and 50-100µm. The authors found that mortar containing 70% glass fine aggregate with a particle sizes of 36-50 µm showed about 80% higher compressive strength and 5% higher flexural strength than the control mortar. This increment of strengths was mainly attributed by the reduction of macropores and mesopores in the microstructure as a result of pozzolanic reaction of fine glass particles. Besides, Batayneh et al. [87] found that specimens with 5-20% glass fine aggregate having particle size ranges 0.15-4.75mm showed about 12-45% higher compressive, 14-43% higher flexural and 16-33% higher tensile strengths than the control specimens. The authors stated that the surface texture and strength of the glass particles might have positive influence on the strength development of glass fine aggregate mixed mortar and concrete. Similarly, Adaway and Wang [88] reported that concrete containing 30% glass fine aggregate with particle sizes of 0.075-1.18 mm showed about 6% higher compressive strength than the control concrete. The authors mentioned that the angular nature of the glass particles improved the bonding between the cement paste and aggregate. As a result, the concrete containing glass fine aggregate showed higher compressive strength than the control concrete. In another study, Ling and Poon [89] found that the use of 15% GGBFS as SCM improved the compressive strength of mortar using 100% glass fine aggregate by about 5.9% with comparable flexural strength to that of the reference mortar at 90 days. On the other hand, some researchers found lower compressive, flexural and tensile strengths for the specimens with glass fine aggregate having particles sizes of 0.075-5 mm as compared to the control specimen [89-90]. This is mainly attributed to (i) the decrease of the overall density of concrete containing glass fine aggregate with higher fineness modulus (FM) [89], (ii) weaker adhesion between the smooth surface of glass particles and cement paste [84], or (iii) the presence of lead in glass that may negatively affect the cement hydration [90]. Furthermore, Soliman and Tagnit-Hamou [91] reported that concrete with 50% fine glass aggregate having a mean particle size of 0.6 mm showed about 5% lower compressive strength than the control concrete due to the elongated and flattened nature of the glass particles. From the above discussion, it can be seen that the influence of particle size of glass fine aggregate in concrete and mortar is contradictory among the researchers due to the wide variation of the surface texture or nature of glass particles. However, it is obvious that smaller ranges of glass fine aggregate (<1 mm) may influence the strength development of concrete and mortar positively, even up to high sand replacement level.

2.3.1.3 Curing age

It was noticed that the curing age and temperature can affect the strength properties of GP blended specimens. Limbachiya [92] found that compressive and flexural strengths of concrete with 15% glass fine aggregate and normal concrete were very close at various ages between 3 days and 365 days. In contrast, Ling and Poon [93] reported that mortars consisted of 100% glass fine aggregate showed about 15% lower compressive strength and about 33.6% lower flexural strength than the control mortar at 90 days. This could be due to the weaker bonding between the smoother surfaces of glass aggregate and cement paste which resulted in the reduction of strengths. Similar observations have been reported by Wright et al. [94] and Ling and Poon [95]. However, Bostanci et al. [96] found that concrete containing 15% glass fine aggregate did not show any noticeable reduction in both compressive and flexural strengths as compared to the control concrete at 365 days. The authors explained that the presence of irregular and angular shaped glass particles increased the contact area of interfacial bond between cement paste and the aggregates. Therefore, the use of broken waste glass as fine aggregate to a certain percentage may provide satisfactory mechanical properties due to its morphological characteristics. In summary, prolonged curing has little or negligible influence on the strength development of glass fine aggregate mixed mortar and concrete. Furthermore, reactive glass fine aggregate may participate in ASR at the later ages, which may affect the strength development negatively.

2.3.1.4 Glass fine aggregate content

Park et al. [81] reported that compressive, tensile and flexural strengths of concrete decreased with the increase of glass fine aggregate content. The authors highlighted several reasons for the decrease of strength of glass fine aggregate concrete including (i) the decrease of adhesion between the glass aggregate surface and cement paste, (ii) the increase in fineness modulus of fine aggregates, and (iii) the decrease of compacting factor. Similarly, Choi et al. [97] reported that mortar containing 25-100% glass fine aggregate showed about 19-38% less compressive and 11-32% less flexural strength than the control mortar at 90 days. Besides, Ali and Al-Tersawy [98] found that the concrete with 10-50% glass fine aggregate exhibited about 4-21% less compressive, 7-29% less flexural and 4-23% less tensile strengths than the control concrete

at 28 days, where the cement content of the mixture was 450 kg/m³. Furthermore, Lam et al. [99] found that concrete paving block consisted of 50% glass fine aggregate and 50% recycled fine aggregate showed about 54 MPa compressive and 3.36 MPa tensile strengths at 28 days, whereas 25% glass fine aggregate with 75% recycled fine aggregate showed about 56 MPa compressive strength and 3.90 MPa tensile strength at the similar age. On the other hand, Wang [100] found that the concrete with 20% glass fine aggregate showed about 99-100% compressive and 98-99% flexural strengths to the control concrete, and about 14% higher tensile strength as compared to the control concrete at 90 days. However, Mardani-Aghabaglou et al. [101] found that concrete made of 15-60% glass fine aggregate exhibited about 2-11% less compressive and 2-15% less tensile strengths than the control concrete. The authors mentioned that glass fine aggregate had lower density, more friability and higher surface smoothness as compared to those of the limestone fine aggregate. As a result, strength decreased with the increase of glass fine aggregate content. However, Sharifi et al. [102] reported that concrete with 10% glass fine aggregate showed about 2% higher flexural and 11% higher tensile strengths as compared to the control concrete at 28 days. In addition, Ismail and Al-Hashmi [103] reported that the concrete with 20% glass aggregate only represented about 4.23% higher compressive strength than the control concrete, whereas the concrete mixes with 10%, 15%, and 20% glass aggregate showed about 3.57%, 6.96%, and 11.20% increase in flexural strength as compared to the control mix. Sharifi et al. [102] claimed that the addition of a little volume of waste glass as fine aggregate provided better adhesion between the aggregate and cement paste that led to an improvement of flexural and tensile strengths. However, the use of a higher volume of glass fine aggregate reduced the bond strength between aggregate and cement paste due to the more smooth surface texture of glass aggregate. Therefore, concrete associated with high amount of glass fine aggregate displayed lower strength than the control concrete. Sikora et al. [104] also observed a similar trend, when 25-100% natural sand was replaced by glass fine aggregate to produce mortar samples. The authors hypothesised that the presence of high content of waste glass aggregate increased the stress concentration that led to a reduction of strength. Furthermore, Romero et al. [105] found that glass fine aggregate concrete showed higher compressive strength than the control concrete for up to 30% replacement level. In another study, Penacho et al. [106] reported that natural sand can be replaced by glass aggregate by up to 50% without negatively affecting the strength properties of mortar. The authors mentioned that pozzolanic potential of some of the very fine glass particles increased the equivalent binder mix content, which improved the strength properties.

2.3.2 Durability properties

2.3.2.1 Water absorption

The water absorptions of mortar and concrete containing glass fine aggregate reported in various published literature are presented in Table 2.6.

	Optimum results			Water absorption			
Source of glass fine	Glass	Particles	Age	Decrease	Increase (%)	- References	
aggitgait	aggregate (%)	size (mm)	(uays)	(70)	(70)		
Waste glass	20	<0.6	56	-	1.19	[85]	
Recycled glass	100	90%<2.36	90	38	-	[89]	
Blue glass bottles	100	<5	90	10.2	-	[93]	
Recycled glass	15	<4	28	-	2.4	[101]	
Waste glass	25	-	-	-	12.5	[104]	
Waste glass	20	23.4%<0.	28	-	7.5	[106]	
		15					
Funnel and bottle glass	100	<5	90	21	-	[107]	
Recycled glass	5	<4	-	-	2	[108]	

Some studies reported that mortar and concrete containing glass fine aggregate showed less water absorption than the control mixtures [89, 93,107]. However, other studies stated that glass fine aggregate mixed mortar and concrete exhibited higher water absorption than the control mortar and concrete [85, 101, 104, 106, 108]. Sikora et al. [104] and Penacho et al. [106] found that water absorption of glass aggregate mixed mortar increased with the increase of sand replacement level. The authors noticed about 12.5% and 7.5% higher water absorptions than the control mortar at 28 days for 25% and 20% sand replacement, respectively. The authors [104] explained that the total porosity of mortar increased significantly when glass fine aggregate was used as a partial replacement of natural sand. As a result, water absorption of the mortar increased substantially. Similar trend was also reported by Patel and Dalal [85] and Mardani-Aghabaglou et al. [101]. Besides, Castro and Brito [108] reported that concrete with 5% glass aggregate showed negligible increase in water absorption (about 2%) as compared to the control concrete. However, some researchers [93,107] found that 100% glass fine aggregate

mortar and concrete showed about 10-21% less water absorption than the reference mixture at 90 days. This was attributed to the lower water absorption capacity of glass particles than sand particles. The authors found that water absorption of river sand was 0.87% whereas, water absorption of glass fine aggregate was nearly zero. Therefore, cementitious mixes containing glass fine aggregate exhibited less water absorption than the control mix. Furthermore, Ling and Poon [89] mentioned that the use of 60% GGBFS as SCM reduced the water absorption of mortar containing 100% glass fine aggregate by 38%.

2.3.2.2 Drying shrinkage

The drying of shrinkage of mortar and concrete containing glass fine aggregate reported in different published studies are presented Table 2.7. It has been noticed that the use of broken waste glass as fine aggregate in mortar and concrete reduced the drying shrinkage significantly.

	Optimum	results		Drying shrinkage		
Source of glass fine aggregate	Glass fine aggregate (%) (%)	Particles size (mm)	Ages (days)	Decrease (%)	References	
LCD glass	20	<4.75	28	40	[77]	
Soda lime glass bottles	75	<4.75	56	11.7	[80]	
Glass bottles	45	<5	112	18.4	[89]	
CRT glass	75	<4.75	112	22.5	[90]	
Recycled glass cullet	100	90%<2.36	56	49.33	[93]	
Recycled glass cullet	100		39	31.8	[94]	
Fluorescent lamp glass	40		112	60.9	[95]	
Recycled glass	15	<5	112	11.11	[96]	
Funnel and bottle glass	100	<5	90	53.3	[107]	
LCD glass	30	<4.75	90	40	[109]	
Soda lime brown glass	100	-	90	17.1	[110]	

Table 2.7 Impact of glass fine aggregate on the drying shrinkage of mortar and concrete

LCD - Liquid crystal display; CRT - Cathode ray tube

Wang et al. [77] reported that concrete containing 20% glass fine aggregate exhibited about 40% less drying shrinkage than the control concrete. Besides, Bostanci et al. [96] and Du and Tan [110] stated that glass fine aggregate mixed high strength concretes (50 MPa and 60 MPa design strength) showed insignificant drying shrinkage. Similarly, Tan and Du [80] found that

mortar with 25-100% glass fine aggregate showed less shrinkage values as compared to control mortar. The authors highlighted that glass fine aggregate mortar samples showed significant resistance against drying shrinkage due to the (i) negligible water absorption capacity of glass particles, (ii) higher aspect ratio, (ii) irregular shape of glass particles and (iv) relatively higher volume fraction of fine aggregate. Furthermore, it was noticed that mortars with 50% or 75% glass fine aggregate showed lower drying shrinkage than the mortar with 100% glass fine aggregate. This was attributed to the presence of higher portion of finer glass particles that brought higher amount of moisture into the system to cause higher shrinkage [80]. Furthermore, Zhao et al. [90] found that concrete with 75% glass fine aggregate showed about 22.5% less drying shrinkage than the control concrete. Besides, Wright et al. [94] observed that concrete with 100% glass fine aggregate showed about 31.8% less drying shrinkage than the control concrete. In addition, the use of 75% GGBFS as SCM reduced the drying shrinkage of 100% glass fine aggregate based mortar about 50% [89]. In another study, Ling and Poon [93] observed that the mortar with 100% glass fine aggregate showed less drying shrinkage up to 56 days and after that the shrinkage values started to increase. The authors found that the mortar with 100% glass fine aggregate showed about 20% higher drying shrinkage than the control mortar at 112 days.

2.3.2.3 Alkali silica reaction (ASR)

There is a debate among researchers regarding the role of glass fine aggregate on the ASR expansion of mortar and concrete [81, 89, 92, 111-112]. The ASR test results of mortar and concrete using glass fine aggregate reported in previous studies are presented in Table 2.8. Some researchers found insignificant ASR expansion in glass fine aggregate mixed mortar [90, 114], whereas some other researchers reported that the use of glass as fine aggregate substantially increased the ASR expansion of mortar and concrete [99, 105]. ASR of glass fine aggregate mortar and concrete depends on several factors including glass type [116-117], particle sizes of glass [82,118-119], quantity of glass fine aggregate [95, 113, 120], and age of the specimen [112, 121-122]. Jin et al. [116] reported that the mortar with 10% clear glass (having particle sizes 0.149 mm or 0.297 mm) showed comparable expansion to that of the control mortar. The authors also mentioned that the mortar with 10% clear glass fine aggregate due to the presence of Cr_2O_3 in green glass. The authors stated that Cr_2O_3 compound present in green glass acted as an ASR inhibitor by forming less expansive gel containing Cr^{3+} . Similarly, Saccani and Bignozzi [117] found that soda-lime glass fine aggregate mixed mortar

showed less expansion than the mortars containing uncoloured borosilicate glass or amber borosilicate glass or lead-silicate glass at 10%, 25% and 35% sand replacement levels. The authors concluded that glass with different colours showed difference in reactivity due to the variation of chemical compositions under alkaline environment. Chen et al. [82] noticed that expansion of mortar containing 20% waste E-glass fine aggregate decreased by approximately 52% due to the pozzolanic effect and lower equivalent alkali content (Na₂O and K₂O) of fine E-glass particles less than 0.3 mm size.

	Optimum	results		Expan		
Source of glass fine aggregate	Glass fine aggregate (%)	Particle s size (mm)	Age (days)	Increase (%)	Decrease (%)	References
Coloured glass	10	0.3-4.75	14	50	-	[81,111]
Waste E-glass	20	< 0.3	16	-	52	[82]
Glass bottles	15	<5	14	20	-	[89]
CRT glass	25	<4.75	28	10	-	[90]
Coloured glass	5	<5	14	14.5	-	[92]
Blue glass bottles	100	2.36-5	28	18	-	[93]
Fluorescent lamp glass	10	-	14	-	460	[95]
Coloured glass	50	-	28	93		[99]
Mixed waste glass	20	<4.75	14	-	66.2	[103]
CRT glass	10	-	14	1150	-	[105]
Waste glass	50	<5	28	33.33	-	[107]
Coloured glass	100	<4	1095	-	15	[112]
Waste glass	25	0.3-4.75	14	4.2	-	[113]
Recycled glass	5	-	28	14.3	-	[114]
Mixed colour glass	50	1.19-	14	-	13.6	[118]
		0.15				
Soda-lime glass	20	0.24	546	-	102.5	[119]
Coloured glass	100	0.3-4.75	28	-	95.7	[121]
Coloured glass	10	0.3-4.75	1095	397	-	[122]
Glass bottles	100	<0.6	14	110	-	[123]
Soda-lime glass	10	0.16-5	14	1000	-	[124]
Coloured glass	100	<2.36	28	-	577	[125-126]

Table 2.8 Effect of glass fine aggregate on the ASR of mortar and concrete

CRT- Cathode ray tube

Similarly, Rajabipour et al. [118] found that the mortar with 50% coloured glass aggregate with particle sizes of 0.149-1.19 mm showed about 13.6% less expansion as compared to control mortar. Similar observations were also reported by Soliman and Tagnit-Hamou [91] and Idir et al. [119] (Table 2.8). The authors [91, 119] stated that glass particles of size less than 1 mm

formed non-expansive local ASR gel around the particles, which introduced a better bonding between the particles and cement paste. As a result, expansion reduced significantly. In addition, Ismail and AL-Hashmi [103] found that a mortar with 10-20% glass fine aggregate of size less than 4.75 mm displayed about 66.2% less expansion than the control specimen. However, some researchers reported that ASR of mortar and concrete increased with the increase of glass fine aggregate content [95, 113, 120]. For example, Topcu et al. [120] reported that the mortar containing 100% white coloured glass fine aggregate showed about 77% higher expansion than the mortar with 25% white coloured fine glass aggregate after 21 days in AMBT. In another study, Zhu et al. [121] stated that 100% glass fine aggregate mixed mortar showed about 95.7% less expansion than the control mortar at 28 days; however, displayed about 200% higher expansion at 133 days as compared to control mortar in AMBT. In concrete prism tests (CPT), Yuksel et al. [122] noticed about four times higher expansion after 3 years for using 10% glass fine aggregate mixed concrete as compared to the control concrete. In contrast, Dhir et al. [112] found that concrete containing 100% glass showed 15% less expansion than the control concrete even after 3 years.

On the other hand, some studies mentioned that the use of glass fine aggregate caused deleterious expansion in mortar due to the high reactive nature of glass particles [93, 107, 123-124]. Therefore, these studies suggested to use different types of SCM or lithium compounds as ASR suppressor in glass fine aggregate cementitious mixes. For instance, Lam et al. [99] recommended to use 10% pulverized FA by weight of total aggregate in concrete in order to control the ASR expansion when glass fine aggregate content is 25% or more. Besides, Topcu et al. [120] suggested that the utilization of 50% FA or 5% lithium carbonate (Li₂CO₃) can effectively suppress the ASR of glass fine aggregate mixed concrete. Furthermore, Du and Tan [110, 125-126] mentioned that ASR of 100% glass fine aggregate mortar can be suppressed effectively by using various amounts of ASR suppressors. Based on their experimental results, the optimum content of various types of ASR suppressors were found as 10-50% FA, 45-60% GGBFS, 12.5% SF, 20% GP and 0.5-2% lithium chloride (LiCl) or Li₂CO₃.

2.3.2.4 Resistance to chloride attack

Most of the studies indicated that the use of glass fine aggregate in mortar and concrete reduced chloride permeability as seen in Table 2.9. Chen et al. [82] found that chloride-ion penetration decreased significantly with the increment of E-glass content of up to 30%. The use of 30% E-glass as replacement of sand reduced the chloride penetration of concrete about 86%. Similarly,

Kou and Poon [89] reported that 45% glass fine aggregate mixed concrete showed about 60% higher resistance to chloride ion penetration than the control concrete. The authors mentioned that lower porosity and better packing efficiency of glass fine aggregate than that of river sand and natural granite increased the chloride resistance significantly. Besides, Wang et al. [77] and Wang [100] reported that the use of glass fine aggregate reduced chloride ion penetration significantly for up to 80% natural sand replacement level. Some researchers noticed that 100% glass fine aggregate mortar and concrete showed about 62-80% higher resistance to chloride attack than the control mortar and concrete [80, 94 110]. Wang and Huang [109] explained that the use of glass fine aggregate reduced chloride ion penetration by filling the pores in concrete. On the other hand, Shayan and Xu [127] reported that concrete with 50% glass fine aggregate showed about 6.12% lower resistance against chloride penetrability than the control concrete. Similarly, Castro and Brito [108] found that concrete with 20% glass fine aggregate displayed about 10% higher chloride penetration depth as compared to control concrete. However, the authors [108] claimed that experimental problems connected with the environmental conditions of the dry chamber and with adjustment of the equipment might be responsible for the negative results of glass fine aggregate mixed samples.

	Optimum results			Chloride pe		
Source of glass fine aggregate	Glass fine aggregate (%)	Particles size (mm)	Age (days)	Increase (%)	Decrease (%)	References
LCD glass	80	<4.75	28	-	79	[77]
Waste E-glass	30	<2.36	-	-	85.9	[82]
Glass bottles	45	<5	28	-	60.2	[89]
Mixed waste glass	100	-	28	-	62	[94]
LCD glass	80	<2.38	-	-	62.5	[100]
Recycled glass	20	<4	28	10	-	[108]
LCD glass	30	<4.75	180	-	13.3	[109]
Coloured glass	100	<4.75	28	-	80	[80, 110]
Waste glass	50	0.15-2.36	380	6.12	-	[127]

Table 2.9 Effect of glass fine aggregate on the chloride resistance of mortar and concrete

LCD- Liquid crystal display

2.3.2.5 Resistance to sulphate attack

Although the number of conducted research on sulphate resistance of concrete and mortar containing glass fine aggregate is limited, the glass fine aggregate concrete and mortar showed satisfactory performance against sulphate attack as presented in Table 2.10 [80, 82, 100]. Wang [100] noticed that concrete with 20-80% glass fine aggregate showed higher resistance against sulphate attack and the weight loss of the concrete due to sulphate attack decreased significantly with the increase of glass fine aggregate content. For instance, the authors found that the concrete with 20% glass fine aggregate showed about 27% lower weight loss as compared to the control concrete, whereas the concrete with 80% glass fine aggregate exhibited about 60% lower weight loss as compared to the control concrete. Based on the microstructural investigations, the authors revealed that the use of glass fine aggregate produced denser microstructure from the well connection between the glass fine aggregates and cement hydration products (C-S-H gel). Therefore, glass fine aggregate concrete showed relatively lower weight loss when exposed to sulphate solution.

	Optin	num results			
Source of glass fine aggregate	ource of Glass ass fine fine Particles ggregate aggregate (mm (%)		– No. of wet-dry cycle	Decrease (%)	– References
Coloured glass	75	<4.75	10	5	[80]
Waste glass	50	0.038-0.3	5	82.3	[82]
LCD glass	80	<2.38	5	30	[100]

Table 2.10 Effect of glass fine aggregate on the sulphate attack of mortar and concrete

LCD- Liquid crystal display

In another study by Tan and Du [80] found that mortars containing 25-100% glass fine aggregate showed comparable weight loss to that of control mortar after 10 cycles of wet-dry sulphate exposures. Furthermore, the authors noticed that glass fine aggregate mortars exhibited significantly higher compressive and flexural strengths than the control mortar after 10 cycles of wet-dry sulphate exposures. From the visual observation, the authors revealed that the surface of glass fine aggregate mortar specimens was attacked by sulphate while the inner core of specimens was intact from the sulphate attack. Besides, the authors also stated that finer glass particles might have participated in pozzolanic reaction at high temperature (oven-drying

at 105°C) of the wet and dry cycles. As a result, mortars with glass fine aggregate exhibited higher resistance against sulphate attack. Similarly, Chen et al. [82] found that concrete with 10-50% E-glass fine aggregate showed substantially lower weight and strength losses than the control concrete after five cycles of wet-dry sulphate exposures. The authors claimed that the use of E-glass as a substitute of natural sand in concrete provided good performance against sulphate attack by minimizing surface defects.

2.4 Use of GP as a partial precursor in alkali activated systems

The previous studies on the application of GP as a partial substitution of FA or GGBFS in preparation of AAC are summarized in Table 2.11. The influence of GP as partial precursor material on the workability, mechanical and durability performance of AAC is briefly described in the subsequent sections.

2.4.1 Workability

Shoaei et al. [131] reported that the workability of alkali activated GGBFS mortars gradually increased with the rise of GGBFS replacement level using GP. The authors noticed that the flow value of mortar increased from 90 mm to 120 mm for replacing 40% GGBFS by GP. In addition, Samarakoon et al. [134] showed that the workability of alkali activated FA-GGBFS mortars was enhanced when 10% to 30% FA was replaced using GP. These studies reported that the inclusion of GP as a partial precursor material promoted the movement of paste matrix due to its smooth characteristic. In contrast, Samadi et al. [136] and Liu et al. [137] reported that the addition of GP as a replacement of FA or GGBFS reduced the flowability of alkali activated FA-GGBFS mortars due to the angularity of GP particles.

2.4.2 Mechanical properties

Bobirica et al. [128] reported that the use of GP as a partial replacement of FA could not bring any improvement on the mechanical strength of AAC even after heat curing. Similarly, Tho-In et al. [129] found that the compressive strength of heat cured alkali activated FA pastes declined from 45.7 MPa to 33.3 MPa for using 40% GP as a substitution of FA. The authors explained that when GP added as a substitution of FA, the silicon (Si) and aluminium (Al) ratio increased remarkably since GP consisted of high Si and very low Al contents. The increase of Si to Al ratio led to the formation of unstable or poor cross-linked reaction products, thus compressive strength reduced. Some studies investigated the mechanical performance of AAC when GP was used as a partial substitution of commercial GGBFS [130, 132-133].

Din den system	GP	Particle sizes of	Optimum	Investigated	Deferences
Binder system	(%)	GP (µm)	GP (%)	properties	References
FA+GP	10-30	<74	10	compressive strength and microstructure	[128]
FA+GP	10-40	<12	10	compressive strength and microstructure	[129]
GGBFS+GP	20-50	23	20	compressive strength, microstructure and ASR	[130]
GGBFS+GP	10-40	25	30	workability, compressive strength, flexural strength, water absorption, ASR and microstructure	[131]
GGBFS+GP	25-75	38	25	workability and compressive strength	[132]
GGBFS+GP	25-50	-	25	compressive strength, permeability properties and microstructure	[133]
FA+GGBFS+GP	10-30	11.5	30	workability, compressive strength and microstructure	[134]
FA+GGBFS+GP	10-30	5.07	30	compressive strength and microstructure	[135]
FA+GGBFS+GP	5-20	-	5	workability, mechanical properties, microstructure	[136]

Table 2.11 Summary of the previous works on the use of GP in alkali activated systems

FA-Fly ash; GGBFS-Ground granulated blast furnace slag; GP-Glass powder; ASR-Alkali silica reaction

These studies reported that the inclusion of GP as a partial replacement of commercial GGBFS led to the reduction of compressive strength of alkali activated GGBFS mortars. The authors explained this phenomenon as the reactivity of GGBFS with alkaline solution was much higher than that of GP. This statement shows inconsistency with the results reported by Shoaei et al. [131]. The authors claimed that the inclusion 10% to 30% GP as a partial replacement of GGBFS enhanced the formation of C-S-H gel, which led to increase in compressive strength

of GGBFS mortars. However, the authors observed a substantial decrease in strength for the further increase of GP content beyond 30%. The authors stated that the replacement of GGBFS at higher percentages using GP resulted a notable decease in Ca/Si ratios, which hindered the generation of C-S-H or calcium aluminium silicate hydrate (C-A-S-H) reaction products. Recently, some studies [134-135] reported that the use of GP as partial replacement of FA in FA-GGBFS blended system showed promising outcomes in terms of the mechanical properties. Zhang et al. [135] observed significant strength gain when 10% to 30% FA was replaced using GP in FA-GGBFS binder systems. Similar observation reported in the study reported by Samarakoon et al. [134]. The authors mentioned that the positive influence of using GP in FA-GGBFS systems was due to the dissolution of more Si and Ca elements from GP, which promoted the formation of aluminosilicate gels such as C-A-S-H and calcium-(sodium) aluminosilicate hydrate (C-(N)-A-S-H). In addition, Samadi et al. [136] observed that the inclusion of 5% GP as a replacement of GGBFS in FA-GGBFS binder system improved the mechanical and microstructural properties significantly. However, the further addition of GP beyond 5% affected the properties negatively. The authors explained that when GGBFS was replaced using GP at a higher replacement ratio, a substantial amount binder particles remained undissolved or unreacted due to the decrease of Ca and Al elements that led to the formation of incomplete or weak three-dimensional framework.

2.4.3 Durability

Some previous studies [130, 131, 133, 137] investigated the durability related properties of FA and GGBFS based alkali activated systems where FA or GGBFS was replaced partially using GP. Liu et al. [137] noticed that the use of 20% GP as a replacement of FA or GGBFS introduced to a higher drying shrinkage at all testing ages. The authors stated that the chemical shrinkage of GP is relatively higher as compared to FA or GGBFS, thus alkali activated systems incorporated GP exhibited higher shrinkage than that of the reference system. However, the authors found that the inclusion of 20% GP in FA-GGBFS binder systems improved the carbonation resistance significantly. The authors claimed that the additional alkalis as released from the dissolution of GP particles enhanced the pore solution alkalinity, which restricted the carbonation occurrence. Besides, Maraghechi et al. [130] and Shoaei et al. [131] reported that the use of GP as a partial replacement of GGBFS reduced the potential ASR activity of reactive aggregate in alkali activated systems. The authors stated that replacing of GGBFS by GP reduced the availability of free Ca element since the amount of calcium oxide (CaO) phase in GP is much lower than that of GGBFS. Consequently, the use of GP as partial

substitution of GGBFS in alkali activated systems reduced the risk of potential ASR in case of reactive aggregate. In addition, some studies [131-133] reported that the inclusion of GP as a partial precursor material showed negative influence on the permeability properties such as water sorption and sorptivity. Shoaei et al. [131] found that the water absorption of alkali activated FA-GGBFS mortars containing 10%, 20%, 30% and 40% were 7.14%, 7.40%, 7.70% and 7.76%, respectively, whereas 6.59% in alkali activated FA-GGBFS mortar without GP. Similar observation was reported by Cercel et al. [133]. The authors mentioned that the larger particle size and lower water absorption capacity of GP particles as compared to that of traditional precursor materials (FA and GGBFS) are primarily responsible for high water absorption of the alkali activated systems containing GP.

2.5 Use of waste glass as fine aggregate in alkali activated systems

The research studies on the use of the waste glass cullet as fine aggregate in FA and GGBFS based AAC are summarized in Table 2.12. The effects waste glass fine aggregate on the workability, mechanical properties and durability performance of AAC are discussed in the following subsections.

2.5.1 Workability

Lu and Poon et al. [138] reported that workability of the alkali activated FA-GGBFS mortars improved substantially when waste glass cullet was used as a substitute of natural sand. The authors noticed that the flow gradually increased with the increase of replacement percentage of natural sand by waste glass cullet. For instance, the flow values of the FA-GGBFS mortars with 25%, 50%, 75% and 100% glass fine aggregate were 117 mm, 124 mm, 132 mm and 137 mm, respectively, whereas the flow value of the FA-GGBFS mortar without glass fine aggregate was 108 mm. The authors mentioned that the positive influence of using waste glass fine aggregate on the workability is attributed to the smooth surface texture and low water absorption capacity of glass fine aggregate. Similarly, Saccani et al. [141] noticed that the workability of alkali activated FA mortars increased from 59.1% to 69.6% when 10% natural sand was replaced using glass fine aggregate. In contrast, Gholampour et al. [139] reported that the use of glass fine aggregate in alkali activated systems reduced the workability remarkably, irrespective of binder type. According to their study, the flow of alkali activated FA mortar containing natural sand was 150%, whereas 85% in alkali activated FA mortar containing glass fine aggregate. Similarly, the flow of alkali activated FA-GGBFS mortar containing natural sand was 100%, whereas 50% in alkali activated FA mortar containing glass fine aggregate.

The authors explained that the particle size and angularity of glass cullet are higher than those of natural sand, therefore, its inclusion as fine aggregate increased the inter-particle friction that led to the reduction of flowability.

Binder system	Glass fine aggregate (%)	Particle sizes (mm)	Optimum glass fine aggregate (%)	Investigated properties	References
FA + GGBFS	25-100	0.15-5	25	workability, compressive	[138]
				strength, fire resistance	
FA + GGBFS	100	maximum size-1.91	100	workability, compressive strength, water absorption, drying shrinkage, microstructure	[139]
FA + GGBFS	100	0.018	100	compressive strength, drying shrinkage, microstructure	[140]
FA	10	0.075-2	10	workability, compressive strength, density, porosity, sulphate resistance, freeze-thaw resistance, alkali silica reaction	[141]
FA + GGBFS	100	0.6-5	100	high temperature resistance	[142]

Table 2.12 Summary of the previ	ous works on the use o	of glass fine aggregate in AAC
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FA-Fly ash; GGBFS-Ground granulated blast furnace slag; GP-Glass powder

2.5.2 Mechanical properties

Lu and Poon et al. [138] found that the compressive strength of alkali activated FA-GGBFS mortars gradually deceased with increase of glass aggregate content. However, an improvement in flexural strength was noticed when 25% to 50% glass fine aggregate was used. The authors stated that glass fine aggregate cannot provide strong bond at the interfacial transition zone (ITZ) due to its smooth surface texture, thus compressive strength reduced. On

the other hand, the angularity and aspect ratio of glass fine aggregate are much higher than that of natural aggregate, which led to the improvement of flexural strength. Besides, Gholampour et al. [139] observed that the compressive strength of alkali activated mortars containing glass fine aggregate was very close as compared to alkali activated mortars with natural sand. Additionally, the authors found that the use of glass fine aggregate improved the tensile strength of alkali activated mortars. In addition, Saccani et al. [141] reported that the use of glass fine aggregate reduced the compressive strength due to its smooth surface texture. Furthermore, Zhang et al. [142] investigated the effects of particle size of glass fine aggregate on the compressive strength of alkali activated mortars. The authors found that the alkali activated mortars prepared with smaller size of glass fine aggregate exhibited higher compressive strength as compared to the mortars fabricated with larger size of glass fine aggregate. The authors stated that the improvement of compressive strength is attributed to the reduction of internal porosity by the small size of glass fine aggregate.

2.5.3 Durability

Gholampour et al. [139] reported that alkali activated mixtures containing glass fine aggregate exhibited much lower water absorption as compared to the alkali activated mixture with natural sand, regardless of binder type and glass aggregate content. This phenomenon was attributed to the lower water absorption of glass fine aggregate than that of natural sand. However, the authors found that the use of waste glass as fine aggregate increased the drying shrinkage of alkali activated systems. Since the water absorption of glass particles is much lower than of sand particles, its inclusion favoured the presence of free water during the reaction process that the led to an increased moisture loss during the drying shrinkage process. On the other hand, Hajimohammadi et al. [140] found that glass fine aggregate based alkali activated mixtures showed higher drying shrinkage than that of alkali activated mixtures with natural sand. The authors stated that glass fine aggregate contains a significant proportion of finer size of glass particles that may have dissolved partially or fully in the presence of alkaline liquid, resulting an increase of the amount of paste content and thus increase of the ultimate shrinkage of the matrix. In addition, Saccani et al. [141] noticed that AAC containing glass fine aggregate exhibited similar stability compared to the AAC with natural sand when subjected to freezethaw cycles and sulphate exposure. Besides, some studies [138, 142] indicated that the presence of waste glass as fine aggregate improved the high temperature resistance of AAC. The authors explained that glass aggregate was partially softened when exposed at high temperatures and this improved the bonding at the ITZ as well as introducing denser structure. Therefore, glass

fine aggregate alkali activated mixtures exhibited excellent resistance against high temperature exposures. Since a significant amount alkaline solutions are commonly used in preparation of AAC and glass fine aggregate contains great amount of reactive silica component, their concurrent use may trigger the risk of ASR. Considering these aspects, some previous studies [139, 143] investigated the ASR susceptibility of glass fine aggregate when used in alkali activated systems. However, the number of studies covering this issue is insufficient and the findings of those previous works are contradictory to each other. For instance, Xie et al. [143] reported that alkali activated FA mortars containing waste glass fine aggregate showed greater than 0.1% expansion in AMBT due to high ASR susceptibility of glass fine aggregate. In contrast, Gholampour et al. [139] found that the AMBT expansions of alkali activated mixtures containing glass fine aggregate were well below than the threshold limit (0.1%).

2.6 Environmental benefits

The reuse of waste glass as a construction material provides several benefits to the environment. Since waste glass is not bio-degradable material, its use in construction sectors will reduce the environmental problems caused at the landfill sites. Besides, the glass manufacturing process requires huge amount of raw materials and thermal energy. Consequently, the consumption of raw material and energy can be reduced significantly if the glass is recycled. Ferdous et al. [144] reported that recycling of each ton of glass minimizes the use of about 19 litres of oil, 42 kilowatt-hour of energy and 1.5 cubic meters of landfill space, and also reduces the release of about 3.4 kilogram of air pollutants. Similarly, Islam et al. [44] claimed that recycling of one ton of glass saves equilvalent amount of natural resources. Furthermore, Imteaz et al. [145] found that the concentration of heavy metals and other contaminting elements present in recycled crushed glass are within the safe limit specified in environment protection authority of Victoria, Australia.

2.7 Limitations and research gaps

This chapter critically reviewed the literature on potential use of waste glass as supplementary binder and aggregate for sustainable cement-based and alkali activated construction materials. Generally, crushing of waste glass requires high energy and long period of time since raw or as-received waste glass is not suitable to use as a part of binder or fine aggregate in Portland cement based systems due to (i) the presence of higher amount of impurities, (ii) the wide ranges of size and shape, (iii) the sharp edges and (iv) the unsuitability of coarser glass particles to participate in pozzolanic reaction. This chapter critically reviewed the mechanical properties

such as compressive, flexural and tensile strengths, and durability related properties including water absorption, drying shrinkage, ASR, and chloride and sulphate resistances of waste glass mixed cement-based and alkali activated composites. It was found that the inclusion of waste glass as a partial precursor material and fine aggregate in the production of alkali activated composites showed promising results in terms of workability, comparable mechanical strength and durability related properties as compared to those of conventional alkali activated composites. However, the available research works related to the utilization of glass as a partial precursor material and fine aggregate in alkali activated system is very limited. The findings of the existing studies emphasized that waste glass has a considerable potential for use as a partial precursor material and fine aggregate in alkali activated systems. Overall, it has been identified that the effects of waste glass as a partial precursor material and fine aggregate has not been studied in alkali activated system as extensively as in cement based systems. Therefore, more research is needed is this area in order to understand the mechanisms more clearly. This study has attempted to fill the gaps, especially to determine the effects of using different percentages of glass as precursor and aggregate on the performance of AAC and understand the underlying mechanisms by microstructural investigations.

2.8 Summary

Literature relevant to the use of waste glass as supplementary binder and aggregate for sustainable cement based and alkali activated based construction materials has been discussed in this chapter. Based on the above discussions, the following points can be summarized:

- In cement based systems, the pozzolanic reactivity of waste glass powder is mainly dependent on its fineness. Besides, the colour of glass also has a significant effect on the reactivity of glass powder due to the variation in chemical compositions among the different coloured waste glasses. In addition, heat curing has a positive influence on the reaction mechanism of glass powder when used as an SCM.
- 2. GP mixed OPC mortar and concrete exhibited significant improvement in compressive, flexural and tensile strengths as compared to those of control mortar and concrete with up to 30% cement replacement when the average particle size of GP was less 45 μm. Besides, the GP blended samples showed about 13-18% less water absorption, 6-23% less drying shrinkage, 30-70% less expansion due to ASR, 15-80% higher chloride resistance and 30-80% higher sulphate resistance as compared to those of control samples at 10-40% cement replacement level.

- 3. The utilization of waste glass cullet with particle sizes less than 1 mm as fine aggregate in OPC mortar and concrete showed comparable or greater compressive, flexural and tensile strengths as compared to control mortar and concrete with up to 50% replacement of natural sand. The specimens containing glass fine aggregate showed significantly lower water absorption, drying shrinkage, chloride and sulphate resistances than the control specimens containing up to 100% replacement of natural sand. The main concern of using glass fine aggregate is ASR, which can be minimised (i) by using various types of SCMs such as FA, GGBFS and SF or (ii) by reducing the particle sizes of glass aggregate to less than 1 mm.
- 4. In alkali activated systems, the use of GP as a partial replacement of FA or GGBFS in binary blended AAC such as FA-GP or GGBFS-GP cannot provide enhancement of hardened properties. However, it has been noticed that the inclusion of GP as partial replacement of FA enhanced the workability, mechanical properties and durability of ambient cured alkali activated FA-GGBFS mixtures.
- 5. Considering the all above aspects as stated in this chapter, the inclusion of waste glass powder as a partial binder in cement based or alkali activated systems offers significant improvements in the mechanical and durability properties. Therefore, waste glass powder can have potential use in the construction projects such as residential or commercial buildings, bridges and pavements. In addition, the literature reviewed reported that cement based or alkali activated systems containing waste glass cullet as fine aggregate may be designed to have comparable mechanical and some durability related properties to that of cement based or alkali activated systems with natural fine aggregate. However, the major technical concern of using waste glass cullet as fine aggregate is ASR since cement based or alkali activated composites containing waste glass fine aggregate was found more prone to ASR expansion compared to traditional cement based or alkali activated composites. The ASR of cement based or alkali activated composites can be minimized successfully by the inclusion of additives, such as FA, GGBFS and waste glass powder. Moreover, waste glass cullet has also the potential to be a suitable replacement for natural fine aggregate in the construction projects such as buildings, bridges and pavements. It may start with low-risk applications such as in concrete footpaths.

2.9 References

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CHAPTER 3

EFFECTS OF WASTE GLASS POWDER ON THE WORKABILITY, COMPRESSIVE STRENGTH AND DURABILITY OF ALKALI ACTIVATED MORTARS

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This chapter investigates the feasibility of using GP as a partial precursor in FA and GGBFS based alkali activated mortars cured at ambient condition. The influence of GP as a partial precursor on the properties such as workability, compressive strength, drying shrinkage, sorptivity, porosity and chloride permeability of alkali activated FA and FA-GGBFS mortars were evaluated. Furthermore, the experimental results were evaluated by a detail microstructural investigation.

3.1 Overview

Production of cement is one of the prime sources of generating greenhouse gases with high energy usage. Numerous studies focused on the potential use of different industrial by-products as supplementary binder materials in order to reduce the emission of greenhouse gases and embodied energy related to the production of cement [1-3]. Geopolymer composite or alkali activated binder was developed to promote the use of cement-less binder for concrete [4]. This type of binders can be produced by the chemical activation of aluminosilicate by-product materials by alkaline liquids. FA and GGBFS are two most common industrial by-products used to produce alkali activated binders [5-6]. The use of other by-products such as SF, RHA and POFA were also studied by different researchers [7-9]. Meanwhile, a huge quantity of discarded waste glass remains unused, the management of which poses a significant environmental challenge due to its non-biodegradable nature. However, waste glass may have potential for use in the production of alkali activated binders since it has an abundance of amorphous silica [10-11]. Several researchers recently attempted to use GP as a source material to produce alternative sodium silicate solution for the fabrication of AAC [12-13]. Besides, in some previous studies GP was used as a partial replacement of traditional precursor materials to produce AAC [14-16]. However, the synthesis of alternative sodium silicate solution using GP is a complex and energy intensive process since it requires thermal treatment [17] and multiple mechanochemical steps [18].

Meanwhile, some recent studies have emphasized that GP can be used effectively as a precursor material in AAC due to its affinity of high dissolution in alkaline media [19-22]. Tho-In et al. [23] found that 10% to 20% replacement of FA by GP slightly improved (4.2%) the 7-day compressive strength of neat FA geopolymer paste cured at 60°C for the first 48 hours. Similarly, Maraghechi et al. [14] found that alkali activated mortars with 80% FA and 20% GP showed about 13% higher compressive strength at 56 days as compared to the mortar without GP. The authors steam cured the specimens at 60 °C until the testing age that may have facilitated dissolution of more silica form the glass. In contrast, Bobirica et al. [24] reported that the use of GP as a partial precursor reduced the compressive strength of FA based geopolymers even though the samples were cured at 60°C for 24 hours.

Furthermore, some previous studies attempted to use GP as partial precursor in FA-GGBFS binder systems. For instance, Zhang et al. [15] studied the effect of GP as a partial replacement of FA in alkali activated FA-GGBFS binder systems in room temperature. The authors concluded that compressive strength increased significantly with the increase of FA replacement by GP. Similar trend was also reported by Samarakoon et al. [19] for FA-GGBFS binders with GP. The increase of strength is attributed to the formation of additional calcium-dominant reaction products. However, the effect of using GP above 30% was not considered in these studies. Besides, these studies used a considerably higher amount of GGBFS content which was 50% of the total binder. This higher amount of GGBFS might hinder the actual reaction or performance of GP in blended systems due to the higher reactivity of GGBFS in alkaline medium. Furthermore, some previous attempts revealed that the replacement of GGBFS by GP cannot bring any improvement on the compressive strength of ambient cured alkali activated mortar due to the low reactivity of GP as compared to that of GGBFS [14, 25-26].

It can be seen from the above review that the specimens in most previous studies were heat cured for a certain duration to promote the dissolution of reactive silica from glass in the alkaline environment. Moreover, the studies on the performance of GP as a partial replacement of FA in alkali activated FA or FA-GGBFS system at ambient temperature are still scarce in the literature. Only a limited number of studies investigated the influence of GP as a partial precursor in alkali activated systems cured at ambient condition. However, a comprehensive study on the developments of strength and microstructure, and the properties related to durability of ambient cured alkali activated binder using GP is necessary in order to understand its effect. Therefore, this chapter investigated the effect of GP as a partial precursor in alkali

activated FA and FA-GGBFS binder systems cured in ambient condition. The properties of FA and FA-GGBFS binders with GP were evaluated by investigating the workability, compressive strength, drying shrinkage, sorptivity, porosity and chloride permeability. In addition, an extensive microstructural investigation was carried out in order to understand the reaction products and related mechanisms through X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscope (SEM) images and energy-dispersive X-ray (EDX) analysis.

3.2 Details of the experimental work

3.2.1 Materials

FA, GGBFS and GP were used as the solid precursors to prepare alkali activated mortars. Waste glass cullet was collected from SKM recycling group of Victoria which was then ground to fine powder using a laboratory ball mill after washing and drying. The grinding duration was selected to five hours after several trials to obtain the desired finesses. The particle size distribution curves of the materials are shown in Fig. 3.1. The percentages of FA, GGBFS and GP smaller than 75 µm were 89.76%, 95.01% and 98.38%, respectively. The Blaine specific surface areas of the FA, GGBFS and GP were 0.380 m²/g, 0.451 m²/g and 0.492 m²/g, respectively. The chemical compositions of FA, GGBFS and GP, as determined by XRF analysis using a Bruker-AXS S4 equipment are given in Table 3.1. The FA is classified as Class F as per ASTM C618 [27] and GGBFS is categorized as neutral since its basicity coefficient, defined by $K_b = (CaO + MgO)/(SiO_2 + Al_2O_3)$ is equal to 1 [28]. As seen in Table 3.1, the main oxide components of GP were SiO_2 (70.61%), Na_2O (12.8%) and CaO (10.9%), whereas those of FA were mainly SiO₂ (60.03%) and Al₂O₃ (22.75%). Besides, the GGBFS consisted of predominantly CaO (41.46%), SiO₂ (32.72%), and Al₂O₃ (13.37%). The XRD results of raw materials are shown in Fig. 3.2. It can be seen that crystalline phases Quartz (SiO₂), Mullite (Al₆Si₂O₁₃) and Maghemite (Fe₂O₃) were detected in FA and gypsum (CaSO₄.2H₂O) in GGBFS, whereas GP was mainly amorphous. The relative amorphous content of FA and GGBFS were 86% and 95.7%, respectively, as evaluated by quantitative XRD (QXRD) measurement using TOPAS software. To perform QXRD, 3 g of the raw material was mixed with 0.3 g of Al₂O₃ as a standard material and analysed in a diffractometer. The SEM images show that FA particles are mainly spherical in shape (Fig. 3.3a), whereas GP particles are angular and irregular in shape with smooth surface textures (Fig. 3.3b). Fig. 3.3

(c) depicts that GGBFS particles are tetrahedron in shape with a wide range of sizes and rough surface textures.

A blend of commercial Na_2SiO_3 solution (silica modulus ratio, Ms = 2) and NaOH solution of 8 molarity was used to activate the precursors. The mass ratio of NaOH solution to Na_2SiO_3 solution was 2.0. The dissolution rate of GP was reported as fairly quick and high at 8 molar concentration of NaOH solution [16, 19]. Therefore, 8 molar NaOH solution was selected in the present study. Locally available silica sand was used as fine aggregate. The fineness modulus and specific gravity of sand were 1.95 and 2.60, respectively. The sand was prepared to saturated surface dry (SSD) condition following the method of ASTM C128 [29].

Constituents	FA	GGBFS	GP
SiO ₂	60.03	32.72	70.61
Al ₂ O ₃	22.75	13.37	1.43
CaO	3.80	41.46	10.90
Fe ₂ O ₃	6.78	0.83	2.49
K ₂ O	1.28	0.30	0.34
MgO	1.29	5.54	0.69
Na ₂ O	0.54	0.27	12.8
P ₂ O ₅	0.89	0.01	0.02
SO ₃	0.25	4.97	0.11
TiO ₂	1.06	0.60	0.06
MnO	0.07	0.20	0.03
SrO	0.05	0.12	0.01
Cr_2O_3	0.01	0.02	0.06
ZnO		0.01	0.01
Loss on ignition	1.15	0.15	0.11

Table 3.1 Chemical compositions of the materials (mass %)



Fig. 3.1 Particle size distributions of the materials



Fig. 3.2 XRD results of the materials



(a) FA



(b) GGBFS



(c) GP Fig. 3.3 SEM images of the materials

3.2.2 Mix proportions and preparation of test specimens

As seen in Table 3.1, GP has a significant amount of SiO₂ as well as some CaO and Na₂O, whereas the main constituents of FA are SiO₂ and Al₂O₃. Therefore, the partial replacement of FA by GP can be expected to affect the reaction mechanism by providing additional SiO_2 , CaO and Na₂O into the system. Based on this hypothesis, FA was replaced by 10% to 40% GP in preparation of alkali activated FA and FA-GGBFS blended mortars. In alkali activated neat FA system, the mixture containing 100% FA was considered as the reference mix and designated as G0F. The mix ID G10F indicates that the mixture was made up with 10% GP and 90% FA. In alkali activated FA-GGBFS blended system, a mixture containing 70% FA and 30% GGBFS was referred to as the reference mixture and designated as G0FS. The mix ID G10FS denotes that the mixture was consisted of 10% GP, 30% GGBFS and 60% FA. The mortar mix proportions and different molar ratios are given in Table 3.2. The molar ratios were calculated by using the chemical compositions of precursors given in Table 3.1. It can be noted that the overall molar ratios in the mixtures such as Si/Al and Na/Al gradually increased with the increase of FA replacement by GP. This is because of GP contains greater amounts of SiO₂ and Na₂O as compared to FA (Table 3.1). The mass ratio of activator liquid to solid precursor materials was kept constant at 0.6 for all the mixtures. A Hobart mixer machine was used to obtain homogeneous mortar mixture. The fresh mortar was poured into the moulds and compacted by casting on a vibrating table. The mortar specimens were cured at an ambient temperature of $20 \pm 3^{\circ}$ C and the relative humidity of $65 \pm 10\%$ until the testing.

Mix	Constituent mix proportions (kg/m ³)						Molar ratios (calculated)			
ID	FA	GGBFS	GP	NH*	SS**	Sand	Si/Al	Na/Si	Na/Al	Ca/Si
G0F	688	0	0	138	276	1100	2.68	0.28	0.76	0.06
G10F	619	0	69	138	276	1100	2.99	0.31	0.93	0.07
G20F	550	0	138	138	276	1100	3.39	0.34	1.15	0.08
G30F	481	0	207	138	276	1100	3.89	0.37	1.42	0.08
G40F	412	0	276	138	276	1100	4.53	0.39	1.78	0.09
G0FS	481	207	0	138	276	1100	2.71	0.32	0.89	0.25
G10FS	412	207	69	138	276	1100	3.08	0.35	1.07	0.26
G20FS	343	207	138	138	276	1100	3.56	0.38	1.35	0.27
G30FS	274	207	207	138	276	1100	4.18	0.41	1.71	0.28
G40FS	205	207	276	138	276	1100	5.04	0.44	2.21	0.29

Table 3.2 Mix proportions of the alkali activated mortars

* 8 molar sodium hydroxide solution; ** Sodium silicate solution

3.2.3 Test methods

Workability of the fresh mortar was evaluated according to ASTM C1437 [30] using a flow table apparatus. Compressive strength of the hardened mortar specimens of 50 mm cube was determined at 7, 14, 28 and 90 days as per ASTM C109 [31] using a MATEST universal testing machine with a loading rate 1.2 kN/sec.

Mortar specimens of 25 mm × 25 mm × 285 mm were used to determine drying shrinkage at 7, 14, 21, 28, 56 and 90 days as per ASTM C157 [32]. Sorptivity of the mortar was determined at 28 days according to ASTM C1585 [33]. Porosity of the mortar was determined at 28 days as per ASTM C642 [34]. The oven-dry mass, saturated mass after immersion, saturated mass after boiling and immersed apparent mass of the specimen were measured in order to calculate porosity. Rapid chloride permeability test (RCPT) was performed according to the ASTM C1202 [35] at 28 days using a Giatec Perma[™] RCPT testing equipment.

To characterize the reaction products, XRD analysis was performed on paste samples at 28 days using a D8 advance (Bruker AXS) instrument. This instrument scanned the samples from 7° to 90° of 20 angle with a scanning rate of 0.5° /min and steps of 0.015° . TGA was performed on paste samples to investigate the thermal stability of the reaction products using a Mettler Toledo TGA instrument. An aluminium oxide crucible with 30 mg of the powdered sample

was placed into the designated area of TGA instrument and heated up to 1000°C in an Argon environment to measure the change of mass and heat flow with the change of temperature.

SEM and EDX examinations were conducted to evaluate the microstructures of hardened mortar samples using a NEON 40EsB (ZEISS) microscopy instrument. An extra sample was prepared from each mix for the purpose of microstructure study. After 28 days of curing, suitable size of samples were cut from the hardened specimen using a small diamond saw. Then, the samples were coated with 10 nm carbon before the SEM and EDX tests. Back scattered electron and an accelerating voltage of 20 kV were used for the SEM images.

3.3 Results and discussion

3.3.1 Workability

The influence of GP on the flow of alkali activated mortars is presented in Figs. 3.4 and 3.5. It can be seen that flow of the FA mortars decreased from 147% to 122% due to 40% FA replacement by GP. Similarly, flow of the FA-GGBFS mortars decreased from 116% to 95% due to 40% replacement of FA by GP. Thus, the flow of alkali activated mortar decreased by the use of both GGBFS and GP as partial replacements of FA.



Fig. 3.4 Flow of the alkali activated FA mortars with GP



Fig. 3.5 Flow of the alkali activated FA-GGBFS mortars with GP

The flow decreased by about 7.1% to 25.5% in FA mortars and by 5.8% to 21.4% in FA-GGBFS mortars due to the use of 10% to 40% GP. Although flow decreased with the increase of GP content, all the mortars were sufficiently workable for proper mixing, placing, compaction and finishing of the specimens. It can also be noted that the sand was used in SSD condition so it would not affect the quantity of free liquid available in the mixture. Consequently, the flow was affected by the morphology and physical characteristics of GP particles. As seen from Fig. 3.3(a), 3.3(b) and 3.3(c), the FA particles were spherical, whereas both GGBFS and GP particles were angular and irregular in shape. Thus, the replacement of FA by GGBFS and GP resulted in the increase of angular particles in the matrix. The presence of angular and irregular particles reduced mobility of the paste matrix. Besides, the liquid demand is increased when FA is replaced by GP since the specific surface area of GP (0.492 m^2/g) was higher than that of FA (0.380 m^2/g). As a result, the alkali activated mortars with GP showed less flow as compared to the mortars without GP. By comparing Figs. 3.4 and 3.5, it can been noticed that the flow values of the FA-GGBFS mortars were lower than those of the FA mortars. This is due to the higher specific area of GGBFS ($0.451 \text{ m}^2/\text{g}$) as compared to FA (0.380 m²/g), which led to an increase of high liquid demand during mixing. Liu et al. [36] noticed that the replacement of 20% FA by GP reduced the flowability of alkali activated

mortars by 6%. Besides, Huseien et al. [37] reported that the use of nano sized GP as a replacement of 5% to 20% GGBFS reduced the flow by 2% to 13%. The authors stated that the presence of finer GP particles increased the specific surface area of the solid binders. Therefore, the solid binders with high specific surface area showed lower workability due to the higher liquid demand. Furthermore, Lu et al. [38] noticed that flow of the OPC mortar reduced when GP was used as a supplementary cementitious material. The reduction of the fluidity of mortars was attributed to the irregular and angular shape of GP particles.

3.3.2 Compressive strength

The compressive strength developments of ambient cured FA and FA-GGBFS mortars with 10% to 40% GP are shown in Figs. 3.6 and 3.7. It can be seen from Fig. 3.6 that compressive strength of the FA mortars decreased with the increase of GP content at all curing ages. This reduction of strength is more noticeable at the early ages (7 and 14 days) than at the later ages (28 and 90 days). For instance, at 7 days, the reductions of compressive strength were 8.1%, 18.2%, 27.3% and 33.7% for the GP contents of 10%, 20%, 30% and 40%, respectively. However, at 28 days, the reductions of compressive strength were 2.5% to 15.8% for using 10% to 40% GP. This trend is consistent with the findings in some previous studies [16, 23]. The strength gain of FA-GP mortars at the later ages is attributed to the dissolution of significant amount of silica from GP in the alkaline medium [39]. Generally, the silica and alumina present in FA react with the alkaline solution and forms stable network of alkaline aluminosilicate gel, which provides the main strength development of the neat FA based alkali activated binders. As seen in Table 3.1, the amount of SiO₂ in both FA and GP are comparable; however, GP contains very low amount of Al₂O₃ (only 1.43%) as compared to the Al₂O₃ content in FA (22.75%). Consequently, the Si/Al ratio increased with the increase of GP as a replacement of FA, as shown in Table 3.2. As a result, the FA-GP blended systems produced aluminosilicate gel with low Al content [39-40], which is considered as a reason for the lower compressive strength of FA-GP mortars as compared to the FA mortars without GP. This hypothesis will be further justified through characterization of the reaction products and microstructure analysis in the later sections. On the other hand, some previous studies reported that alkali activated mortars produced using binary blends of FA and GP showed higher compressive strength as compared to those without GP [14, 23, 41]. These studies claimed that pH of the pore solution increased substantially when FA was replaced partially by GP to produce alkali activated composites since GP contains a significant amount of Na₂O (usually

more than 10%). This high pH of the pore solution improved the reaction network by adding more silicon from the silica dissolution of the precursors [42]. However, those specimens were heat cured as compared to ambient curing used in this study. The silica dissolution of GP by the alkaline solution at room temperature is usually low [12, 43]. Besides, it is well known that the reaction rate of FA is also relatively slow at ambient temperature. Consequently, the initial reaction kinetics are prolonged with the increase of GP as a partial replacement of FA. Furthermore, it can be observed that the porosity of FA mortars gradually increased with the increase of GP content as reported in section 3.3.5 and thus resulted in the decrease in compressive strength with the increase of GP content.



Fig. 3.6 Compressive strength development of the alkali activated FA mortars with GP



Fig. 3.7 Compressive strength development of the alkali activated FA-GGBFS mortars with GP

Fig. 3.7 shows that compressive strength of the FA-GGBFS mortars slightly improved when 10% to 20% FA was replaced by GP. This improvement is significant at the later ages as compared to that at early ages. The mortars containing 10% and 20% GP showed 3.3% and 5.5% higher strengths, respectively, as compared to the mortars without GP at 7 days. However, the mortars containing 10% and 20% GP exhibited 5.3% and 11.3% higher strengths, respectively, as compared to reference mortar at 90 days. This is attributed to the addition of more silicon ions into the aluminosilicate network supplied by GP [19]. Besides, the finer GP particles also reduced porosity due to the filler effect as discussed in section 3.3.5 However, it can be noticed that compressive strength declined when the replacement of FA by GP exceeded 20% in the FA-GGBFS blended alkali activated mortars. Similarly, Zhang et al. [15] and Bobirica et al. [24] found that the partial replacement of FA by GP in FA-GGBFS alkali activated binders resulted in an improvement of compressive strength up to a certain level of GP replacement rate and then the strength declined beyond that level. Generally, when alkali activated composites are cured at room temperature, the use of GGBFS promoted the initial reaction kinetics due to the presence of higher amount of CaO in GGBFS [44]. Thus, the partial

replacement of FA by GP offers an improvement in compressive strength of FA-GGBFS blended mortars unlike the compressive strength trend as observed in the neat FA mortars in Fig. 3.7. Besides, GGBFS contains 13.37% Al₂O₃ as seen in Table 3.1, therefore, the use of a constant percentage of GGBFS (30%) may compensate the reduction of Al₂O₃ due to the replacement of 10% to 20% FA by GP. However, when 30% or more FA was replaced by GP, the Si/Al and Na/Al ratio increased as seen in Table 3.2 due to the decrease of Al₂O₃ and increase of SiO₂ and Na₂O, which affected the reaction mechanism. It is usually accepted that strength of alkali activated composites increases with the increase of Si/Al and Na/Al ratios up to a certain value and then it declines with the further increase of these ratios [45-46]. This was due to an excess amount of Si or Na ions that weakens the chemical network to reduce the mechanical strength [47-48]. In the present study, the optimum values of Si/Al and Na/Al ratios were found to be 3.56 and 1.35, respectively. This is in good agreement with the results reported by Juengsuwattananon et al. [48] and De Silva et al. [49].

3.3.3 Drying Shrinkage

Drying shrinkage values of the FA and FA-GGBFS mortars with 10% to 40% GP are shown in Figs. 3.8 and 3.9. It can be observed that the drying shrinkage values of FA-GGBFS mortars were lower than those of FA mortars. The decrease of drying shrinkage is attributed to the presence of a substantial amount of GGBFS (30%) in the FA-GGBFS mortars that contributes to form C-S-H gel. Production of the C-S-H gel reduced the evaporation and related shrinkage by binding the excess water of the matrix [50]. Besides, in the FA-GGBFS mortars, the presence of GGBFS reduced the interconnection among capillary pores due to its high affinity to the alkaline solution [51]. Some previous studies also reported similar phenomenon [50, 52].



Curing Period (Days)

Fig. 3.8 Drying shrinkage of the alkali activated FA mortars with GP

It can be observed from Fig. 3.8 that the inclusion of GP as a partial precursor resulted in an increase of drying shrinkage in the FA-GP system. For instance, drying shrinkage of the FA mortar without GP was 4040 microstrains, whereas, shrinkage values of FA mortars with 10%, 20%, 30% and 40% GP were 4300, 4800, 5590 and 5920 microstrains, respectively. Similarly, drying shrinkage of the FA-GGBFS mortars increased with the increase of GP content as shown in Fig. 3.9. This is attributed to the availability of free water in the capillary network due to the low water absorption capacity of GP [53]. Furthermore, the chemical compositions of the precursors have a vital role on the drying shrinkage of alkali activated materials. It has been reported that FA based alkali activated specimens are associated with a greater amount of Na₂O showing a higher drying shrinkage of the alkali activated specimens increased with increase of Ca/Si ratio in the source materials. Referring to Tables 3.1 and 3.2, it can be noted that Ca/Si ratio in the mixes increased with the increase of GP content since GP contains higher percentages of CaO and SiO₂ than FA. As a result, alkali activated FA and FA-GGBFS mortars

without GP showed lower drying shrinkage as compared to the mortars containing various percentages of GP.



Fig. 3.9 Drying shrinkage of the alkali activated FA-GGBFS mortars with GP

3.3.4 Sorptivity

Sorptivity coefficients of the alkali activated FA and FA-GGBFS mortars with various percentages of GP are presented in Figs. 3.10 and 3.11. As seen from the Fig. 3.10, the sorptivity coefficient increased gradually with the increase of the percentage of GP in FA mortars. The trend is attributed to the slow reaction rate of FA in alkali activated systems at ambient temperature [55] which is further reduced by the inclusion of GP due to the tendency of its slower reaction at ambient conditions [43]. Therefore, the combination of FA and GP as precursors in ambient cured alkali activated systems may introduce higher amount of unreacted particles due to poor or incomplete reaction that causes high porosity as reported in section 3.3.5. As a result, the addition of GP increased the sorptivity coefficient of the FA mortars. This observation may appear to be different the results reported by Deb et al. [56], which

showed that the addition of 2% nano-silica significantly reduced the sorptivity coefficient of ambient cured alkali activated mortars. This is attributed to the higher reactivity and percentages of finer particles of nano-silica as compared to GP.



Fig. 3.10 Sorptivity coefficients of the alkali activated FA mortars with GP

Interestingly, it has been noticed that sorptivity coefficient significantly reduced when 10% to 20% FA was replaced by GP in the FA-GGBFS mortars as shown in Fig. 3.11. However, sorptivity coefficient increased with further increase of GP content beyond 20%. This supports the observation of Huseien et al. [37] which reported that the use of 5% to 10% GP decreased water absorption of FA-GGBFS mortars by forming denser C-A-S-H gel. Moreover, the inclusion of 10% to 20% GP as a replacement of FA in FA-GGBFS systems may reduce porosity by the filling effect of GP particles as well as by producing some additional reaction products along with the main reaction compounds. However, when the GP content exceeded 20%, it may increase porosity by increasing the presence of unreacted GP particles. Overall, the sorptivity results show consistency with the porosity results reported in section 3.3.5 and microstructural observations as reported in section 3.3.9.



Fig. 3.11 Sorptivity coefficients of the alkali activated FA-GGBFS mortars with GP

3.3.5 Porosity

Porosity values of the alkali activated FA and FA-GGBFS mortars with different percentages of GP are given in Figs. 3.12 and 3.13. It is seen Fig. 3.12 from that porosity of the FA mortars increased with the increase of GP content. On the other hand, as seen in Fig. 3.13, 10% and 20% replacements of FA by GP decreased the porosity of FA-GGBFS mortars. It can also be noted that the FA-GGBFS mortars with 30% GP showed slightly lower porosity than the reference mortar. The porosity results are in good correlation with compressive strengths and sorptivity coefficients of the mortars as presented in the earlier sections as well as those reported in previous studies [57-58]. Moreover, it is noticeable that replacing FA by different percentages GP in the FA based system reduced the reaction rate in ambient curing condition. This slow reaction process left higher amounts of partially reacted or unreacted particles that increased porosity with the increase of GP content in the FA mortars. However, the use of 10% and 20% GP reduced the porosity of FA-GGBFS mortars significantly. Generally, the initial reaction activity is mostly triggered by the GGBFS in the FA-GGBFS systems due to its high reactivity (relative amorphous content of GGBFS is 95.7%). When GP was added in the FA-GGBFS systems, it helped to produce a solid and dense reaction network by providing additional silicon. Consequently, FA-GGBFS mortars with 10% to 20% GP showed low porosity values despite their high drying shrinkage as reported in section 3.3.3. The porosity results will be further explained by the microstructural changes of mortars in the later sections.



Mix ID

Fig. 3.12 Porosity of the alkali activated FA mortars with GP



Fig. 3.13 Porosity of the alkali activated FA-GGBFS mortars with GP

3.3.6 Chloride permeability

Chloride permeability of the alkali activated FA and FA-GGBFS mortars with various percentages of GP was determined by RCPT method and the results are presented in Figs. 3.14 and 3.15. Most of the RCPT devices are designed with the maximum safe current limit of 500 mA and the standard test duration of RCPT is 6 hours as per ASTM C1202 [35]. It has been noticed that the passing current through the alkali activated FA mortar specimens reached the maximum safe limit of RCPT device (500 mA) before 6 hours. Similar observations were also reported in previous studies [59-61], where RCPT was adopted to determine the chloride permeability of FA based alkali activated specimens. The RCPT device stopped performing automatically once it experienced 500 mA of current, therefore, it was not possible to evaluate chloride permeability of the FA mortar specimens by knowing the total charge passing after 6 hours of the testing period. As a result, alternatively, the test duration before reaching to 500 mA of current was recorded to assess the chloride resistance of the FA mortars with GP as shown in Fig. 3.14. It can be noticed that the duration of the test decreased with the increase of GP content. Therefore, it can be said that the passing of chloride ions increased with the increase of GP content in the FA mortars is associated with the GP content. The alkalinity of pore solution is considered as a key factor for passing charges in RCPT [62]. It can be expected that when FA was partially replaced by GP, the considerable proportion of alkaline activator may remain unused even after 28 days of curing due to the slow reactivity of both GP and FA at ambient temperature. Besides, GP contributes alkalis in the pore solution during the activation since GP contains about 13% of alkali oxides (Table 3.1). Consequently, the pore solution alkalinity of FA mortars containing GP increased with the increase of GP content. In addition, as discussed in the section 3.3.5, it can be noticed that the porosity of the FA mortars gradually increased with the increase of FA replacement by GP. As a result, the FA mortar mixtures with GP allowed to pass higher chloride ions in RCPT in a shorter time due to the presence of significant amount of free alkalis in the pore solution. Furthermore, the inclusion of GP increased porosity of the FA mortars as discussed in section 3.3.5, which can be considered as another reason for the higher conductivity of FA and GP blended systems.



Fig. 3.14 RCPT results of the alkali activated FA mortars with GP

As seen from Fig. 3.15, RCPT could be continued for 6 hours without exceeding the safe current limit for the FA-GGBFS mortar specimens. This is attributed to the presence of substantial amount of GGBFS content (30%) in FA-GGBFS mortars which reduced the availability of free alkalis in the pore solution by forming stable reaction products. Besides, it can be noted that the total charge passed decreased when 10% to 20% FA was replaced by GP in the FA-GGBFS mortars. However, further increase of GP content beyond 20% resulted in an increase of the charge passed. For example, the total charge passed through the G20FS and G40FS specimens were 3727 Coulombs and 4935 Coulombs, respectively, whereas the total charge passed through the GOFS specimen was 4288 Coulombs. The positive influence of using 10% to 20% GP on the RCPT results of FA-GGBFS mortars is related to the consumption of more alkalis from GP during the formation of aluminosilicate gels as well as reduction of porosity as reported in the section 3.3.5. In the FA-GGFBS mortars, the reaction product is mainly C-A-S-H gel, whereas N-A-S-H gel is typically formed in FA based system. The alkali binding capacity of C-A-S-H gel is significantly higher than the N-A-S-H gel [63]. Therefore, when 10% to 20% GP was added in the FA-GGBFS system, the alkalis provided by GP were mostly consumed by the reaction product of C-A-S-H gel. However, the pore solution alkalinity

may be increased when 30% to 40% FA was replaced by GP due to the presence of an excess amount of alkalis from the GP.



Fig. 3.15 RCPT results of the alkali activated FA-GGBFS mortars with GP

3.3.7 XRD analysis

The XRD results of alkali activated FA and FA-GGBFS pastes with various percentages of GP is presented in Fig. 3.16. As seen in Fig. 3.16, Quartz and Mullite are the main phases detected in G0F, G20F and G40F paste samples. These phases mainly belong to the raw FA as shown in Fig. 3.2. Besides, the detection of aluminosilicate product phase was difficult due to its amorphous nature. The presence of crystalline Zeolite and Gismondine phases represent the formation of N-A-S-H and C-A-S-H gels, respectively as reported in previous studies [64-65]. Generally, the intensity of peak for zeolite phase is more prominent in the heat cured FA samples than those of ambient cured samples due to the higher degree of reaction. For this reason, relatively smaller peak of zeolite phase was detected at 16.5° of 2θ in the G0F sample. The intensity of this peak gradually decreased with the increase of FA replacement by GP. This



indicates the formation of relatively low amount of N-A-S-H gel in G20F and G40F as compared to the G0F sample.

Fig. 3.16 XRD results of the alkali activated FA and FA-GGBFS pastes with GP

Besides, very small peak for Gismondine can be seen in both G20F and G40F samples at 44.9° of 20. It is likely that the presence of calcium present in GP that may have reacted with the available alumina and silica to form C-A-S-H or C-(N)-A-S-H gels. However, the peak intensity of the Quartz and Mullite phases are relatively high in G20F and G40F samples compared to G0F sample that indicated the presence of more unreacted FA particles in these binder systems.

The XRD results of G0FS, G20FS and G40FS paste samples indicate the presence of Quartz, Mullite and calcium dominant phases such as Gismondine (C-A-S-H) and C-S-H. The primary hydration compound in these binder systems is C-A-S-H, which is coexisted with C-S-H as Gismondine phase in between 20 of 29° and 32°. The intensity of this phase were higher in G20FS and G40FS samples as compared to G0FS. This is attributed to the increase of silicon and calcium elements with the increase of GP content. The evidence of Gismondine and Zeolite peaks along with C-S-H in G20FS indicates the formation of substantial amount of reaction products in this binder system. On the other hand, zeolite phase is not observed in the G40FS sample. The binder compositions of this sample is associated with low aluminium and high sodium contents as compared to G0FS and G20FS as shown in Table 3.2. Consequently, most of the available aluminium ion may have been consumed in the formation of C-A-S-H gel, whereas the available sodium ion may be existed in C-(N)-A-S-H gel. The XRD results of the study show consistency with the XRD results reported by Zhang et al. [15] and Samarakoon et al. [19].

3.3.8 TGA-DTA analysis

The TGA-DTA results of alkali activated FA and FA-GGBFS pastes with various percentages of GP are presented in Figs. 3.17 (a) to 3.17 (f). The figures show the changes in heat flow and mass loss of the samples with the increase of temperature as obtained from the TGA experiments. As seen from the DTA curves of the Figs. 3.17 (a-f), multiple endothermic peaks were formed in the temperature ranges of 100°C to 200°C, 600°C to 700°C and 800°C to 900°C. The formation of endothermic peak between 100°C and 300°C is related to the evaporation of the physically and chemically bound water [15, 64]. Therefore, the highest mass loss occurred in this temperature range as seen in Figs. 3.17 (a-f). When the temperature exceeded to 300°C, the mass loss occurred due to the decomposition of hydroxide and metal ions from the reaction product [66]. This continues up to 600°C and the further mass loss beyond 600°C is due to the decomposition of calcium based compounds [67]. Therefore, the mass loss in the temperature range between 300°C and 600°C can be used to understand the relative degree of reaction for alkali activated composites since the hydroxide and metals ions present in the C-A-S-H or N-A-S-H gel are decomposed in this temperature regime [64]. The higher mass loss in this temperature range corresponded to the existence of greater amount of C-A-S-H or N-A-S-H gel in the matrix. Fig 3.17 (c) shows that sample G40F experienced the lowest mass loss, whereas sample G20FS exhibited the highest mass loss in this temperature regime (Fig. 3.17e). Therefore, the highest mass loss in sample G20FS indicates the formation of greater amount of reaction compounds in that sample. Similarly, the G40F sample showed the lowest mass loss due to the presence of lower amount of reaction products. Similarly, Nergis et al. [68] reported that alkali activated neat FA sample showed greater mass loss in

TGA as compared to the sample with 70% FA and 30% GP between 300°C and 600°C. Overall, the TGA-DTA results show good agreement with the XRD results as presented in the section 3.3.7.



(a) GOF paste sample



(b) G20F paste sample



(c) G40F paste sample



(d) G0FS paste sample



(e) G20FS paste sample



(f) G40FS paste sample

Fig. 3.17 TGA-DTA analysis of the alkali activated FA and FA-GGBFS pastes with GP

3.3.9 SEM and EDX examinations

The SEM images of mortar samples G0F, G20F and G40F are presented in Figs. 3.18, 3.19 and 3.20, respectively. As seen in Fig. 3.18 (a), the unreacted or partially reacted FA particles are embedded by semi homogeneous gel. The EDX of this gel shows high peaks for Si, Al and Na elements as shown in Fig. 3.18 (b). The Si/Al, Si/Na and Na/Al ratios of the gel formed in sample G0F are 2.4, 0.18 and 0.44, respectively, which indicates the formation of N-A-S-H gel in this binder system [69]. By comparing Figs. 3.19 (a) and 3.20 (a) with Fig. 3.18 (a), it can be noticed that the appearance of the unreacted particles are considerably higher in both G20F and G40F mortars as compared to G0F mortar. Besides, some micro cracks can be seen in the microstructure of G20F and G40F mortars. These micro cracks may be generated due to the poor connectivity of the reaction products. Besides, such micro crackes can be developed by high drying shrinkage (refer to Section 3.3.3) and self-desiccation process. EDX at the gel produced in G20F and G40F mortars also traced the peaks for Na, Al and Si elements as like in GOF sample, as seen in Figs. 3.19 (b) and 3.20 (b). It can also be noted that the ratios of Si/Al, Si/Na and Na/Al are higher in the gel formed in G20F and G40F samples as compared to those of GOF sample, as shown in Fig. 3.21 (a). However, in the FA-GP systems, compressive strength gradually deceased with the increase of GP content. This phenomenon is attributed to the significant reduction of alumina with the increase of GP content since the alumina content plays an important role to produce a stable polymer network [70]. As a result, partial replacement of FA by GP reduced compressive strength of the alkali activated FA mortars as presented in Fig. 3.21 (a).



(a)



(b)

Fig. 3.18 SEM and EDX spectra of G0F mortar sample



(a)



(b)

Fig. 3.19 SEM and EDX spectra of G20F mortar sample



(a)



(b)

Fig. 3.20 SEM and EDX spectra of G40F mortar sample

The SEM images of GOFS, G20FS and G40FS mortars are presented in Figs. 3.22, 3.23 and 3.24, respectively. It can be seen from Fig. 3.23 (a) that the microstructure of the G20FS is densely compacted with less pores or visible cracks as compared to the microstructures of GOFS and G40FS as seen in Figs. 3.22 (a) and 3.24 (a). It is also observed from Fig. 3.23 (a) that the unreacted particles are well connected with the reaction products in G20FS mortar. Besides, the filling effect of unreacted GP may improve the pore refinement of G20FS mortar (Fig. 3.23a). Similarly, the microstructure of the G40FS mortar is more dense and compacted; however, it exhibited significant number of micro cracks (Fig. 3.24a). The formation of these cracks may be caused by the presence of excess amount of GP that hinders the formation of reaction products with crosslinking structures. As seen in Figs. 3.22 (b) and 3.23 (b), the intensity of identified peaks at the reaction products of G0FS and G20FS mortars are apparently similar. However, Fig. 3.21 (b) shows that the molar ratios such as Si/Al and Ca/Si are higher in the gel formed in G20FS samples as compared to G0FS sample. This indicates the presence of GP in G20FS mortar assisted to form additional aluminosilicate gels such as N-A-S-H and C-A-S-H gels by contributing more Si and Ca elements. This observation shows a good agreement with the XRD results as reported in the earlier section. As a result, G20FS mortar showed higher compressive strength than the GOFS mortar. Fig. 3.21 (b) shows that the reaction product of G40FS mortar is associated with high Si/Al and Ca/Si ratios; however, the intensity of Al peak is relatively low as seen in Fig. 3.24 (b). Consequently, it can be said that C-S-H is mainly formed in the binder system of G40FS mortar instead of C-A-S-H or N-A-S-H gel due to the lack of sufficient Al. These observations show good relationship with the XRD results presented in section 3.3.7 as well as with the microstructure studies reported by Samarakoon et al. [19].


Fig. 3.21 Relation between molar ratios and compressive strength (a) FA mortars with GP (b) FA-GGBFS mortars with GP



(a)



Fig. 3.22 SEM and EDX spectra of G0FS mortar sample



(a)



Fig. 3.23 SEM and EDX spectra of G20FS mortar sample



(a)



Fig. 3.24 SEM and EDX spectra of G40FS mortar sample

3.4 Summary

The experimental results presented in this chapter are summarized as follows:

- 1. The use of 10% to 40% GP reduced the flow by 7.1% to 25.5% in the alkali activated FA mortars and 5.8% to 21.4% in FA-GGBFS mortars. This is attributed to the angular shape and high specific surface of GP particles as compared to the spherical fly ash particles.
- 2. The inclusion of GP as a partial precursor did not bring any improvement to the compressive strength of FA mortars. However, the use of 10% to 20% GP improved compressive strength of the FA-GGBFS mortars by 5% to 11%. This is attributed to the supply of additional Si, Na and Ca elements by GP which improved the polymerization. However, the use of GP dosage higher than 20% resulted in a decrease of the aluminium content, which affected the polymerization and thus reduced compressive strength.
- 3. The use of GP as a partial replacement of FA caused increase of drying shrinkage in both FA and FA-GGBFS mortars. This is attributed to the increase of Na/Si and Ca/Si ratios which enhanced moisture movement in the aluminosilicate compounds. Furthermore, the slow dissolution and impermeable nature of GP increased the availability of evaporable water in the capillarity networks.
- 4. The use of 10% to 20% GP in FA-GGBFS mortars reduced the sorptivity, porosity and chloride permeability by the improvements of reaction mechanism and reduction of connectivity between the capillary pores.
- 5. Microstructures investigation revealed that the inclusion of GP in FA based system delayed the reaction kinetics due to the lower dissolution tendency of both FA and GP particles at ambient temperature. However, additional C-S-H and C-A-S-H gels are formed in the FA-GGBFS binder system due to the use of GP which produced a compact and dense microstructure.

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CHAPTER 4

ACID RESISTANCE OF ALKALI ACTIVATED MORTARS CONTAINING WASTE GLASS POWDER

The contents presented in this chapter is submitted to the Journal of Cleaner Engineering and Technology for possible publication, (Under review).

This chapter examines the efficacy of GP as a partial precursor in alkali activated mortars exposed to sulphuric acid (H_2SO_4) and hydrochloric acid (HCl) solutions for one year. The changes in physical appearance, surface alkalinity, mass, compressive strength and microstructure of the hardened samples after immersion in acid solutions were evaluated.

4.1 Overview

A few studies are available in the literature that reports the acid resistance performance of AAC containing GP [1-3]. Previously, Vafaei et al. [1] and Vafaei and Allahverdi [2] investigated the acid resistance of geopolymer mortar incorporated with 76% GP and 24% calcium aluminate cement (CAC). The authors reported that GP and CAC blended geopolymer mortars showed much higher acid resistance than cement mortars. Recently, Idir et al. [3] studied durability including resistance to acid, sulphate, carbonation and alkali silica reaction of AAC containing three different types of GP. The authors considered GP as a sole precursor for the production of AAC and reported the findings in terms of glass types. Meanwhile, literature indicate that the inclusion of certain amount of GP as a partial replacement of traditional precursor material such as FA and GGBFS can lead to the improvement in the hardened properties of AAC [4-5]. However, the available studies in this topic covered mostly mechanical and microstructural properties, whereas the technical information related to durability concern such as acid resistance is still insufficient. Therefore, it is essential to evaluate the efficacy of GP as a partial precursor on the durability of AAC since concrete structures built in marine, mining and industrial areas frequently experience aggressive acidic surroundings during their service periods [6-7]. To date, there is no study in the literature that highlights the viability of GP as substitution of traditional precursors on the acid resistance performance of AAC. As seen in the Chapter 3, the inclusion of GP as a partial replacement of the FA in FA based AAC did not bring significant improvements on the hardened properties such as compressive strength, drying shrinkage and chloride permeability, whereas such properties significantly improved when GP was used as a partial precursor in FA and GGBFS blended AAC. Considering this improvement, the influence of GP as a partial precursor

material on the acid resistance of FA and GGBFS blended AAC is investigated in this chapter. For this purpose, FA was substituted by GP at various rates to prepare the samples and the hardened samples were exposed in both H₂SO₄ and HCl solutions for one year. The effects of GP content are evaluated in terms of the changes of visual appearance, surface alkalinity, mass, compressive strength and microstructure of the samples.

4.2 Experimental details

4.2.1 Materials and mix proportions

The materials used in this study were Class F FA, GGBFS, GP, sodium hydroxide solution, sodium silicate solution and silica sand. FA was substituted by GP at 10% to 40% and the amount GGBFS was kept constant at 30% of total binder. A mix without GP was used as the reference to compare the performances of the mixtures containing various percentages of GP. For example, mass percentages of FA, GGBFS and GP were 70%, 30% and 0%, respectively in G0FS mix, whereas those of mix G10FS were 60%, 30% and 10%, respectively. The proportions of the mix constituents are given in Table 4.1. The sodium silicate liquid was mixed with the sodium hydroxide solution at a mass ratio of 2:1 prior to mixing with the precursors. The activator liquid to solid precursor mass ratio was fixed at 0.6 in all mixes. At first, all the solid constituents of the mix were placed in a Hobart mixture and mixed gently at a low speed. Afterwards, the alkaline solution was added and mixed again at a medium speed. Then, the mortar mixture was poured into the 50 mm cube moulds and vibrated using vibrating table to exclude the air bubbles. The demoulded mortar specimens were wrapped using thin plastic film to prevent moisture loss during curing. The specimens were cured in an ambient environment (temperature of $20 \pm 3^{\circ}$ C and relative humidity of $65 \pm 10\%$) for 28 days.

Mix ID	Constituents (kg/m ³)					
	FA	GGBFS	GP	NH*	SS**	Sand
G0FS	481	207	0	138	276	1100
G10FS	412	207	69	138	276	1100
G20FS	343	207	138	138	276	1100
G30FS	274	207	207	138	276	1100
G40FS	205	207	276	138	276	1100

Table 4.1 Mix proportions of the alkali activated mortars

* 8 molar sodium hydroxide solution; ** Sodium silicate solution

4.2.2 Acid exposure procedure

The acid resistance of the mortar samples was evaluated according to ASTM C267 [8]. Initially, the compressive strength and weight of the samples were measured after 28 days of ambient curing. The test procedure followed for the determination of compressive strength is discussed in the section 3.2.3. These values were considered as reference to calculate the percentage strength and weight loss of the studied mortar samples when exposed in acidic media. Thereafter, the mortar samples were immersed in 3% H₂SO₄ and 3% HCl solution separately in plastic containers. The containers were kept at room temperature and the lids of the containers were tightly closed to avoid evaporation. The concentration of the solutions were adjusted after every 30 days by measuring the pH of the solution to keep a steady exposure environment throughout the experimental period. The compressive strength and weight of the samples were determined after 30, 90, 180, 270 and 365 days of acid exposure.

4.2.3 Visual inspection and surface alkalinity measurement

The mortar samples were visually inspected after each designated exposure interval to identify any external cracks, spalling or deterioration due to acid attack. The surface alkalinity of the acid exposed samples were determined by spraying 1% phenolphthalein solution on the crosssection of the samples. For this purpose, the samples were taken out from the acid solutions and cut vertically using a diamond saw. Then, the cross section of the samples were placed under a running tap for 2 to 3 minutes and wiped with a dry cloth. After that, 1% phenolphthalein solution was sprayed on the cross section of the samples and the change of colour of the cross section was monitored. Usually, the phenolphthalein solution remained colourless in the area where pH level is less than 8.3 and changed to pink colour where pH level is greater than 8.3. Therefore, the colourless area of the cross section is considered as a degraded area due to the ingress of acid solution. This procedure was also used to evaluate the apparent surface alkalinity of the acid exposed samples in some previous studies [9-10].

4.2.4 Microstructural examination

The change in microstructure after acid exposure was studied by XRD, SEM and EDX techniques. For the purpose of XRD, paste specimens were prepared and exposed at the same conditions used for mortar specimens. This is because the XRD of paste is more meaningful as it excludes the abundance of the aggregate of mortar specimens. During XRD analysis, the paste samples were finely ground and scanned from 7° to 90° in two theta angle with a scanning

rate of 0.5° /min using a D8 advance (Bruker AXS) instrument. SEM and EDX examinations were conducted using a NEON 40EsB (ZEISS) microscopy instrument. At the end of designated exposure period, a suitable size of samples were cut from the mortar cube specimen using a small diamond saw. Then, the samples were coated with 10 nm carbon before the SEM and EDX tests. The SEM images were captured using secondary electron (SE) with an accelerating voltage of 5 kV and back scattered electron (BSE) with an accelerating voltage of 15 kV.

4.3 Results and discussion

4.3.1 Physical changes and surface alkalinity

Figs. 4.1 and 4.2 show the physical appearance of the mortar samples after 1-year exposure in H₂SO₄ and HCl acid solutions, respectively. As seen in Fig. 4.1, all samples experienced significant deterioration at the edges along with deposition of yellowish-white substances on the surfaces after H₂SO₄ acid exposure. Some visible cracks were found at the edges of G0FS, G30FS and G40FS samples. However, such cracks were not seen in the samples associated with 10% to 20% GP (G10FS and G20FS). Meanwhile, Fig. 4.2 depicts that the samples submerged in HCl solution did not exhibit significant physical damage except some minor changes in the texture of the surface due to deterioration.

The change in surface alkalinity due to the acid exposure as determined by spraying 1% phenolphthalein solution on the cross-section of the acid exposed samples, are presented in Figs. 4.3 and 4.4. It can be noticed that the unexposed sample shows dark pink surface whereas the acid exposed samples exhibited light pink coloured core and a colourless outer layer regardless of type of acid solution. This is due to ingress of the acid solution through the pores during exposure time that reduced the internal alkalinity. However, a significant variation related to perimeter of coloured core among the exposed samples can be noticed. For instance, cross sections of G10FS and G20FS samples show higher perimeter of coloured core than the G0FS sample. On the other hand, cross sections of G30FS and G40FS samples depicted small areas of coloured cores with extended colourless outer layers. Besides, G30FS and G40FS samples exhibited a significant amount of large pores on the surface after the exposure compared to those seen in G10FS and G20FS samples. This clearly demonstrates that the surface alkalinity in G10FS and G20FS samples was less suffered when exposed in the acid solutions due to the ingression of less amount of acid solutions through the pores. Consequently, G10FS and G20FS samples showed less physical damage as compared to the

other samples under similar acid exposure conditions, as shown in Figs. 4.3 and 4.4. These observations show good correlation with the others durability related properties of the studies mortars, as reported in sections 3.3.3 to 3.3.6.



Fig. 4.1 Physical appearance of the mortar samples exposed to 3% H₂SO₄ for one year



Fig. 4.2 Physical appearance of the mortar samples exposed to 3% HCL for one year



Fig. 4.3 Cross sections of the H₂SO₄ exposed samples sprayed with phenolphthalein solution (colourless edges-Degraded area; coloured core-Un-degraded area)



Fig. 4.4 Cross sections of the HCl exposed samples sprayed with phenolphthalein solution (colourless edges-Degraded area; coloured core-Un-degraded area)

4.3.2 Change in mass

The mass change of the mortar samples after exposure to 3% H₂SO₄ and 3% HCl acid solutions at various exposure periods up to 1-year are presented in the Figs. 4.5 and 4.6. An increase of mass was noticed after 28 days in all samples with a range of 0.4% to 0.8% for both types of acid solutions. This is similar to the observations reported by Kwasny et al. [11] and Valencia-Saavedra et al. [12] that alkali activated composites gained slight mass initially after exposure to acid solutions. When the samples were submerged into the acid solutions, it penetrated through the pores of the samples and formed expansive salts. These salts were deposited into the pores of samples without causing any stress at the initial stage [11-12], which increased the mass of the samples. It was found that the mass gain was very close among the G0FS, G10FS and G20FS samples; however, higher mass gains were recorded for the G30FS and G40FS samples. For instance, the increase of mass was 0.3% in G20FS sample and 0.8% in G40FS samples after 28 days exposure in 3% H₂SO₄. This indicates that the use of GP beyond 20% in alkali activated composite resulted in increase of porosity (refer to section 3.3.5) which allows ingress of more solution into the matrix and hence increased the mass. After the initial mass gain, the samples experienced gradual decease of mass with the increase of exposure time. This mass loss was more prominent for exposure to 3% H₂SO₄ compared to the mass loss for exposure to 3% HCl solution. The mass loss of the samples after 1-year exposure to 3% H₂SO₄ was 13.2% to 16.8%, whereas that for 3% HCl varied from 9.1% to 10.9%. This observation is very consistent with the results of mass loss of the alkali activated samples after H₂SO₄ and HCl acid exposures, as reported by Vafaei and Allahverdi [13]. The significant mass loss of the samples by H₂SO₄ acid exposure is due to the formation of a significant amount gypsum which led to decrease of mass by cracking and related deterioration, as shown in Fig. 4.5. It can be noticed that the samples with 10% to 20% GP showed relatively lower mass loss than the sample without GP after exposed to both the acid solutions. However, the samples with 30% and 40% GP exhibited higher mass losses than the samples without GP. Consequently, it is concluded that the positive influence of using 10% to 20% GP against mass loss by acid attack is attributed to the pore filling effect of GP which limits the penetration of acid solution into the matrix during the exposure period. This observation is in support with physical appearance and surface alkalinity of the studied mortars, as discussed in the section 4.3.1. Besides, the change in mass results are in agreement with the soprtivity, porosity, and chloride permeability results of the studied mortars, as discussed in Section 3.3.4, 3.3.5 and 3.3.6. Moreover, the use of 10% to 20% GP as a partial precursor may have modified the reaction

products which assisted to restrict the acute mass loss by acid attack that will be discussed in the later sections.



Fig. 4.5 Mass changes of the mortar samples exposed in 3% H₂SO₄ solution up to one year



Fig. 4.6 Mass changes of the mortar samples exposed in 3% HCl solution up to one year

4.3.3 Change of compressive strength

The compressive strength and porosity of the 28 days cured samples before acid exposure are presented in Table 4.2 which are sourced from in the section 3.3.2. Compressive strength of the reference mix increased by 6% to 10% for using 10% to 20% GP as a replacement of FA. Besides, the mix containing 30% GP showed comparable strength to that of the reference mix. However, compressive strength decreased sharply when the GP content was increased to 40%. The improvement of 28-day compressive strength for using 10% to 20% GP is attributed to the presence of significant amount of SiO₂ (70.61%), Na₂O (12.8%) and CaO (10.90%) in GP, which promoted the polymerization process by providing additional Si, Na and Ca ions. However, the use of high dosages of GP (>20%) reduced the availability of sufficient Al for a favourable polymerization since GP consisted of very low amount of Al_2O_3 (1.43%). In addition, the use of GP dosage beyond 20% increased the porosity as discussed in the section 3.3.5. Consequently, strength dropped when higher percentages of GP were used. The trend observed in the present study is consistent with the results of some recently published studies [4, 14]. The reason for strength gain of the alkali activated composites containing 10% to 20% GP are discussed in Chapter 3 with a detailed microstructural study. This chapter focused on evaluation of the effect of acid attack on the strength of AAM containing GP.

Mix ID	Compressive strength (MPa)
G0FS	71.1
G10FS	75.5
G20FS	78.2
G30FS	67.8
G40FS	62.5

Table 4.2 28-day Compressive strength of the mortars prior to acid exposures

The change of compressive strength of the mortar samples after exposure to 3% H₂SO₄ and 3% HCl acid solutions at various exposure periods until 1-year are presented in the Figs. 4.7 and 4.8. As seen in Figs. 4.7 and 4.8, all samples exhibited substantial loss of strength under both the acid exposure conditions and the strength loss increased sharply with the extension of exposure period. The strength losses of the samples after 3% H₂SO₄ acid exposure were more acute than those immersed in 3% HCl acid solution. The strength loss of the samples varied from 22.2% to 40.1% when immersed in 3% H₂SO₄ solution for 1-year, whereas it varied from

13.3% to 27.6% for immersion in 3% HCl solution for 1-year. This is in line with the observations of Vafaei and Allahverdi [2, 13] and Ridha et al. [15] that reported higher strength loss for exposure to H₂SO₄ than for exposure to HCl solution. As seen in Figs. 4.7 and 4.8, it can be noticed that the strength loss of the reference sample reduced effectively by the inclusion of 10% to 20% GP as a substitute of FA. For example, the samples with 20% GP lost 22.2% and 13.3% of its initial strength after 1-year exposure in 3% H₂SO₄ and 3% HCl solutions, respectively, while the mortar samples without GP lost 31.2% and 21.9% strength at the similar exposure conditions. However, strength loss increased when FA replacement by GP was increased beyond 20%. It has been reported extensively that the strength loss of the alkali activated binder system mainly occurred due to the collapse of aluminosilicate gel network in presence of acidic environment [16-18]. The positive influence of using 10% to 20% GP against strength loss due to acid attack is attributed to the pore filling effect of GP which limits the penetration of acid solution into the matrix during the exposure period. These findings are consistent with the sorptivity, porosity and chloride permeability results as discussed in the sections 3.3.4, 3.3.5, 3.3.6, 4.3.1 and 4.3.2. Besides, the additional Si, Na and Ca element as released from the GP may strengthen the network of the aluminosilicate gels during initial polymerization stage. However, an excess amount of GP introduced agglomeration of undissolved or unreacted GP particles which retarded the progress of aluminosilicate phases as well as causing substantial microcracks [4, 14]. These microcracks helped to extend the penetration area of acid solutions and thus caused severe deterioration. Furthermore, the inclusion of high percentages of GP (>20%) as a substitute of FA may have increased the availability of Ca element since CaO content is higher in GP than that of FA that favoured the formation of gypsum crystals. Consequently, the samples with 30% and 40% GP showed high strength loss when immersed in acid solutions. These hypotheses are discussed through a detailed microstructural examination in the later sections.



Fig. 4.7 The change of compressive strength of the mortar samples exposed in 3% H₂SO₄ solution for one year



Fig. 4.8 Compressive strength changes of the mortar samples exposed to 3% HCl solution for one year

4.3.4 Change in microstructural composition and morphology

4.3.4.1 XRD analysis

The formation of reaction products before and after the immersion in acid solutions are characterized by XRD technique, as shown in Figs. 4.9 and 4.10. As seen in Fig. 4.9 and 4.10, crystalline quartz, mullite and calcium-based compounds such as calcium silicate hydrate (C-S-H), calcium alumino silicate hydrate (C-A-S-H) are the main detected phases of the samples before exposure to acid solutions. Usually, quartz and mullite phases are traced from the unreacted or un-hydrated FA particles that is present in the hardened sample, whereas C-S-H and C-A-S-H are the product of hydration reactions. C-A-S-H phase is coexisted with the primary reaction compound C-S-H (Ca_{1.5}SiO_{3.5}×H₂O) as gismondine (CaAl₂Si₂O8.4H₂O). The trace of zeolite phase (Na₈(AlSiO₄)6(OH)-24H₂O) at around 16-17° of 20 supports the presence of sodium aluminosilicate hydrate (N-A-S-H); however, the intensity of this phase was very low. It can be seen that G20FS shows the higher intensity of C-S-H peak and lower intensity

of un-hydrated phases (quartz and mullite) as compared to GOFS sample. This indicates the additional Si and Ca elements released from GP enhanced the formation of primary reaction products by reducing the availability of un-hydrated components. However, the intensity of the un-hydrated phases increased notably in G40FS. This phenomenon manifested that a significant amount of particles remained un-hydrated or undissolved and produced less amount of reaction products when 40% GP was used as a partial precursor. The XRD results of the present study support the 28-day compressive strength of the mortar samples presented in Table 4.2 and were consistent with some previous studies [4-5].



Legends:

Q-Quartz; M-Mullite; CSH- Calcium Silicate Hydrate; CASH- Calcium Alumino Silicate Hydrate; Z-Zeolite; G-Gypsum

Fig. 4.9 XRD results of the FA-GGBFS paste samples with GP before and after immersion in 3% H₂SO₄ solution

After exposure to 3% H₂SO₄ solution, a new crystalline gypsum phase (CaSO₄.2H₂O) along with the crystalline phases of un-hydrated substances (quartz and mullite) are traced in all the samples as presented in Fig. 4.9. The positions of the hydrated phases (C-S-H, C-A-S-H and N-A-S-H) as detected in the original samples are replaced by the crystalline phases of gypsum,

quartz and mullite after 3% H₂SO₄ acid exposure. This significant alternation of reaction products after immersion in H₂SO₄ solution clearly demonstrates that the H⁺ ions from H₂SO₄ solution weakened the framework of hydrated phases and the HSO₄⁻ ions triggered the formation of crystalline gypsum by reacting with the available Ca^{2+} released from the precursor materials [19-20]. It can be observed that the intensities of gypsum, quartz and mullite phases are significantly lower in G20FS sample than those observed in G0FS sample. This indicates that the Si, Ca, or Na ions from GP are mostly consumed during initial reaction to form stable framework of aluminosilicate gels. Therefore, there was less amount of available Ca ions into the matrix of G20FS mix to form expansive gypsum crystals when exposed in H₂SO₄ solution. As a result, G20FS mix showed the lowest mass loss and strength loss after exposure to H₂SO₄ solution. However, the intensities of these peaks are significantly higher in G40FS as compared to both G0FS and G20FS samples. This is probably due to further dissolution of the unreacted GP particles which adds a substantial amount of Ca²⁺ions into the matrix since GP contains about 10.90% CaO. When a high dosage of GP was used as a partial precursor to fabricate ambient cured alkali activated matrix a significant amount of GP particles remained undissolved in alkaline solution at the initial stage of reaction due to its slow dissolution rate at ambient condition [21]. Later, the cluster of undissolved GP particles increased the porosity of the matrix due to its irregular and smooth surface texture, as discussed in section 3.3.5 [4, 14]. As a result, the hardened samples containing high GP content allowed access of acid solution into the matrix and hence caused a significant deterioration. This supports the losses of mass and compressive strength of the studied mortars after acid exposure presented in the sections 4.3.2 and 4.3.3.



Legends:

Q-Quartz; M-Mullite; CSH- Calcium Silicate Hydrate; CASH- Calcium Alumino Silicate Hydrate; Z-Zeolite

Fig. 4.10 XRD results of the FA-GGBFS paste samples with GP before and after immersion in 3% HCl solution.

Meanwhile, Fig. 4.10 depicts that the phases identified in the original samples remain same after immersion in 3% HCl solution. However, some minor changes related to the position and intensity of the peaks can be observed. For instance, the presence of crystalline quartz and mullite phases increased after HCl exposure. In addition, the intensities of these peak are relatively higher than those detected in original sample. This may due to the increase of the availability of crystalline elements from the precipitation of depolymerized reaction compounds when subjected to acidic environment. Furthermore, C-S-H and C-A-S-H (coexisted with C-S-H) peaks became steep after HCl acid exposure which appeared with broad hump before the acid exposure. This shows that the crystallinity of reaction products (C-S-H or C-A-S-H) increased while exposed to acid solution due to the destruction of polymeric structure [22]. However, after the HCl exposure, the intensities of the peaks observed in G0FS and G40FS samples. This observation indicates that the presence 20% GP limit the deterioration of gel network from HCl acid attack. The reason

for the better performance of the mix containing 20% GP against HCl attack compared to those with higher GP content is same as that for H_2SO_4 attack as discussed above.

4.3.4.2 SEM and EDX examinations

The microstructural changes of the samples after exposure to H_2SO_4 and HCl solutions for 1 year, as investigated by SEM and EDX analysis are presented in Figs. 4.11-4.13. The SEM images reveal that the microstructure of the acid exposed samples consisted of significant cracks and un-hydrated or unreacted particles, as shown in Fig. 4.11 (a-f). The appearance of significant cracks is due to the collapse of aluminosilicate network in presence of acidic agent which led to disintegration of the bonding in the matrix [23-24]. The difference in microstructural damage between the H₂SO₄ exposed samples and HCl exposed samples was apparently minor in low magnification scale since all the samples show substantial cracks under both acid exposure conditions, as shown in Fig. 4.11 (a-f). Therefore, some additional SEM images were captured and analysed in high magnification scale to uncover the influence of GP as a precursor in the matrix when exposed in the acid media, as presented in Figs. 4.12 and 4.13. As seen in Figs. 4.12 (a-c), the paste matrix of G20FS sample is less deteriorated with a more compact structure as compared to the paste matrix of GOFS samples after the H₂SO₄ exposure. Referring to Figs. 4.12 (a) and (b), cluster of rod shaped gypsum crystals can be observed in G0FS sample after H₂SO₄ exposure, whereas the appearance of gypsum crystals in G20FS sample can hardly be seen. However, the trace of sulphur element with low intensity at the binder matrix of G20FS indicates that gypsum crystals may be formed in a limited extent in this matrix. Meanwhile, the microstructure of G40FS sample depicts disintegrated paste matrix with significant amount of undissolved or unreacted GP particles after the H₂SO₄ exposure (Fig. 4.12c). EDX analysis at the paste matrix of G40FS sample confirms the presence of relatively high intensity of sulphur peak as compared to those formed in GOFS and G20FS samples, as shown in Figs. 4.12 (d-f). This clearly reveals that an increased amount of gypsum formed in G40FS matrix, which agree with the XRD results as reported in the previous section. Besides, it can be observed that the intensity of Si, Na and Ca elements are higher in the GP added samples (G20FS and G40FS) than that of the sample without GP content (G0FS). This is due to the presence of higher percentages of SiO₂, CaO and Na₂O contents in GP than in FA. Therefore, when a certain percentage of GP is added as a replacement of FA that improves the reaction mechanism by providing additional Si, Na and Ca elements into the system [25-26]. Besides, the remaining undissolved or unreacted GP particles may serve as a filling agent, which restricted the ingress of H₂SO₄ solution into the system. Furthermore, it can be noted that

the paste matrix of GP added samples (G20FS and G40FS) consisted of lower trace of Al peak as compared to the paste matrix of reference sample. This is because of low Al₂O₃ content of GP than in FA. Therefore, it can be said that the binder matrix of GP added sample is susceptible to form ettringite from the further reaction with gypsum due to lack of sufficient Al element [1-2]. However, the inclusion of an excess amount of GP (>20%) led to increase of undissolved or unreacted GP particles. These undissolved glass particles formed microcracks and connected loosely, which led to increase of porosity in the binder matrix due to its smooth surface texture, as discussed in the section 3.3.5 [27-28]. As a result, the samples with 40% GP (G40FS) showed significantly lower resistance against H₂SO₄ attack than those with 20% GP (G20FS). After one year in H2SO4 exposure After one year in HCl exposure (a) G0FS (d) G0FS (e) G20FS (b) G20FS (c) G40 FS (f) G40FS

Fig. 4.11 Microstructures of the samples after one year exposure in H_2SO_4 (left) and HCl

(right)



Fig. 4.12 SEM and EDX analysis of the samples after one year exposure in H₂SO₄ solution



Fig. 4.13 SEM and EDX analysis of the samples after one year immersion in HCl solution

Meanwhile, as seen in Figs. 4.13 (a-c), the binder phase or paste matrix of the reported mortars was relatively less damaged after immersion in HCl solution compared to those exposed in H₂SO₄. However, the paste matrix appeared to be discontinuous and granular when exposed to HCl solution. This phenomenon is due to the distortion of solid aluminosilicate network by the HCl attack that formed diamond shaped crystalline insoluble precipitation. Interestingly, it can be noticed that the presence of these crystalline precipitations are significantly lower in the G20FS sample as compared to those observed in G0FS and G40FS samples. Besides, it can be seen that the unreacted particles were found to be closely attached with the reaction product in G20FS. On the other hand, the microstructures of G0FS and G40FS samples were found to be more porous and less dense since the deteriorated gel clusters were loosely connected with unreacted particles. The elemental traces and their intensities at the binder phase of the HCl exposed specimens were apparently similar to that of H₂SO₄ exposed samples excluding the notable trace of sulphur element in the H₂SO₄ exposed samples, as shown in Figs 4.13 (d-f). Generally, the highly soluble and detrimental calcium chloride (CaCl₂) salt formed when the samples were immersed in HCl solution, which is primarily responsible for the strength loss of HCl exposed samples [29]. The formation of CaCl₂ is highly dependent on the availability of free Ca element in the pore solution [30]. The primary source of free Ca as supplied from further hydration of GGBFS should be indifferent in all the mixes since the percentage of GGBFS content was constant. However, when higher dosages of GP (>20%) were used as substitution of FA the availability of Ca element increased substantially since GP contained a higher percentage of CaO than FA. Besides, the additional number of undissolved or unreacted GP particles increased the amount of pores in the matrix instead of improving the pore system due to its angular shape. On the other hand, a significant reduction in the amount of pores was seen in the G20FS specimen. These observations show consistency with the porosity results of hardened samples as reported in the section 3.3.5 in Chapter 3. Besides, it is evident from the XRD results that the use of 20% GP led to an enhancement of reaction product. This additional product densified the microstructure by reducing the number of pores [4-14]. Consequently, the G20FS sample showed lower compressive strength loss than G0FS and G40FS samples when immersed in HCl solution.

4.4 Summary

The experimental results presented in this chapter are summarized below:

1. The alkali activated mortar samples containing 10% and 20% GP as a substitution of FA experienced minimal physical damage after immersion in the acid solutions compared to the mortar specimens without GP. However, the mortars with 30% and 40% GP showed more physical damage by acid exposures as compared to the reference mortar. This is because 10-20% GP reduced the porosity due to its pore filling effect of GP which limits the penetration of acid solution into the matrix during the exposure period. However, the use of an excess amount of GP (>20%) increased the porosity which allows for penetration of more acid solution during the immersion period and hence caused physical damages.

2. The use of 10% to 20% GP as a partial precursor reduced the mass and compressive strength losses due to acid attack effectively under both acid exposure conditions. After 1 year of exposure in H_2SO_4 acid, the mortar containing 20% GP showed 13.2% mass loss and 22.1% strength loss, whereas the mass and strength losses of the reference mortar were 14.1% and 31.2%, respectively. In the case of 1 year exposure in HCl acid, 9.1% mass loss and 10.6% strength loss were observed for the mortars with 20% GP, while the control mortar exhibited 13.3% mass loss and 27.6% strength loss. The mass and strength losses increased with the increase of GP content above 20% for both types of acid exposure. This is attributed to the contribution of additional reaction products and pore filling effect of GP up to a certain dosage. Consequently, the acid resistance of mortars significantly improved with the inclusion of 10% to 20% GP.

3. Microstructural examinations using XRD, SEM and EDX analysis confirmed that H_2SO_4 exposed mortar samples experienced higher deterioration than HCl exposed mortar samples due to the formation of gypsum crystals. The use of 10% to 20% GP as a partial precursor densified the microstructure by the improvement of the polymerization mechanism, which reduced the ingress of acid solutions into the matrix. This is due to the release of additional Si, Na and Ca into the system by GP. Consequently, the samples with 10% to 20% GP experienced less deterioration compared to the control samples when immersed in acid solutions. However, the use of higher dosages of GP (>20%) increased the availability of free Ca element as well as porosity due to its angular and irregular shape that increased the deterioration due to acid attack.
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CHAPTER 5

INFLUENCE OF WASTE GLASS FINE AGGREGATE ON THE WORKABILITY, COMPRESSIVE STRENGTH AND DURABILITY OF ALKALI ACTIVATED MORTARS

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This chapter evaluates the properties of ambient cured alkali activated FA and GGBFS blended mortars containing waste glass fine aggregate. The properties include workability, compressive strength, drying shrinkage, sorptivity, porosity, chloride permeability and resistance to high temperature exposures were determined. Furthermore, a detail microstructural examination was conducted to explain the experimental results.

5.1 Overview

Generally, construction industry consumes a huge amount of ordinary Portland cement (OPC) and natural sand to produce concrete. Manufacturing of OPC is responsible for about 6 to 7% of total global CO₂ emissions and it is a very energy intensive process. Since there is a rapid growth in infrastructures and residential development, the contribution of construction sector to global greenhouse gas emission and energy consumption is also increasing [1-2]. Geopolymers or alkali activated binders were developed to reduce the CO₂ emissions and energy consumption of manufacturing OPC by using various supplementary cementitious materials such as GGBFS, FA and SF [3-4]. Recent research revealed that alkali activated mortar and concrete showed better or equivalent mechanical properties and durability compared to those of conventional OPC based mortar and concrete [5-6].

Meanwhile, excessive and uncontrolled use of natural sand in construction industry has introduced serious concern regarding the equilibrium of natural resources and aquatic ecosystems. Consequently, some previous studies emphasized the potential use of various waste products such as ferronickel slag, waste glass and ceramic waste as alternatives to natural sand in cementitious mixtures [7-9]. It has been noticed that the number of research investigating the feasibility of using waste glass as substitution of natural fine aggregate has been increasing during the past few years [10-11]. This is because waste glass recycling industries cannot recycle all types of waste glass due to wide variation of melting point as well

as high recycling cost [12]. As a result, a great amount of waste glass has been used for landfilling since many years. However, landfilling by using waste glass is not environmentally friendly due to its non-biodegradable nature [13].

Considering the above aspects, synthesis of AAC using waste glass cullet is considered as a potential way to reduce the negative environmental effects related to the use of OPC and natural sand. Therefore, some researchers attempted to study the compatibility of waste glass as replacement of natural fine aggregate in AAC in the recent years [14-16]. For example, Lu and Poon [14] investigated the workability, compressive strength, flexural strength and fire resistance of sodium hydroxide (NaOH) activated GGBFS and FA mortars, where 25% to 100% natural sand was replaced by waste glass aggregate. However, the authors did not include microstructural studies and other important properties such as sorptivity, porosity, drying shrinkage and chloride permeability in their study. Besides, the authors performed the high temperature exposure test when the samples were exposed only at 800°C. In another study, Gholampour et al. [15] focused mainly on the comparative performance of waste glass and lead smelter slag as alternatives of natural sand on the mechanical and durability related properties of geopolymer composites associated with various percentages of GGBFS and FA. Recently, Saccani et al. [16] investigated the effect of 10% waste glass as fine aggregate on the durability properties of alkali activated FA mortar. Furthermore, Hajimohammadi et al. [17-18] discussed the influence of fine waste glass powder (median particle size 18 µm) as full replacement of natural sand on the reaction kinetics of alkali activated FA-GGBFS mortars. However, production of glass powder by grinding waste glass cullet is energy intensive and time consuming. Besides, some researchers recommended that the use of glass powder as precursor material for making AAC is more advantageous and suitable due to its chemical compositions [19-20]. Furthermore, our recent research demonstrated that waste glass aggregate can be used in alkali activated FA-GGBFS mortars without causing any deleterious alkali silica reaction (ASR) [21]. This supports further research into the use of glass aggregate in alkali activated mortar since its use in OPC mortar is restricted due to the susceptibility of ASR.

Overall, it is obvious from the previous research attempts that waste glass may have huge potential to be used as a natural sand replacement in preparation of AAC. However, the findings of the existing research is not sufficient to conclude the effectiveness of waste glass aggregate with this type of binder systems without evaluating some important properties such as sorptivity, porosity and drying shrinkage for using waste glass aggregate. Therefore, the current study is designed to investigate the effect of using waste glass fine aggregate on the properties of alkali activated FA-GGBFS mortars based on the research gaps of the existing studies. This chapter evaluates the influence of using 25% to 100% waste glass cullet as a substitution of natural sand on the properties of alkali activated FA-GGBFS mortars. The properties of the alkali activated FA-GGBFS mortars containing waste glass fine aggregate is evaluated by investigating workability, compressive strength, drying shrinkage, sorptivity, porosity, chloride permeability, resistance to high temperature exposures and the microstructures.

5.2 Experimental details

5.2.1 Materials

FA and GGBFS were used as the primary source of aluminosilicate materials to produce alkali activated FA-GGBFS mortars. Table 5.1 shows the chemical compositions of these materials. As per ASTM C618 [22], the FA used in this study can be categorized as Class F since the summation of pozzolanic oxides $SiO_2 + Al_2O_3 + Fe_2O_3$ was over 70% and the amount of CaO was below 10%. The hydration modulus and basicity coefficient of the GGBFS were 1.01 and 1.85, respectively. The loss on ignition of FA and GGBFS were 1.15 and 0.15, respectively. The combination of commercial sodium silicate solution and 8 molar sodium hydroxide (NaOH) was used as the alkaline activator. The mass percentages of SiO₂, Na₂O and H₂O of the sodium silicate solution were 29.4%, 14.7% and 55.9%, respectively. Natural silica sand and waste glass cullet were used as fine aggregate. Natural sand (excavated and processed at sand pits, gravel pits and hard rock quarries) was obtained from Hanson cement group, Perth, Australia. Waste glass cullet was collected from a commercial waste glass recycling company in Australia. The physical properties of natural sand and waste glass cullet are shown in Table 5.2. It can be seen that the glass aggregate had lower density and water absorption capacity as compared to natural sand. However, the fineness modulus of the waste glass was relatively higher than that of natural sand. The physical appearances of sand and waste glass cullet are shown in Fig. 5.1. The waste glass cullet is a mixture of broken glass particles of different colour with varying particle sizes, whereas sand is consisted of white and yellow grains of uniform size. The SEM images of sand and waste glass cullet revealed that the sand particles were sub-rounded and sub-angular in shape with rough surface texture, whereas the glass particles were irregular and angular with smooth surface texture, as shown in Fig. 5.2. The particle size gradations of natural sand and waste glass cullet are presented in Fig. 5.3. Fig. 5.3 indicates that waste glass particles were coarser than the sand. The maximum size of waste glass and natural sand were 2.36 mm and 1.18 mm, respectively. Fig. 5.3 also indicates that

grading of the 100% waste glass exceeded the lower limit of ASTM C33 [23], whilst 100% natural sand exceeded the higher limit of ASTM C33 [23]. However, the grading of the combination of 50% waste glass and 50% natural sand or 75% waste glass and 25% natural sand met the upper and lower limits for fine aggregate as recommended in ASTM C33 [23]. Furthermore, it can be noted that grading of the natural sand and waste glass used in this study satisfied the upper and lower limits for fine aggregate as specified in AS 2758.1 [24]. The aggregates were prepared to saturated surface dry (SSD) condition.

Constituents	FA	GGBFS
SiO ₂	60.03	32.72
Al ₂ O ₃	22.75	13.37
CaO	3.80	41.46
Fe ₂ O ₃	6.78	0.83
K ₂ O	1.28	0.30
MgO	1.29	5.54
Na ₂ O	0.54	0.27
P ₂ O ₅	0.89	0.01
SO ₃	0.25	4.97
TiO ₂	1.06	0.60
MnO	0.07	0.20
SrO	0.05	0.12
Cr ₂ O ₃	0.01	0.02
ZnO		0.01

Table 5.1 Chemical compositions of FA and GGBFS

 Table 5.2 Physical properties of natural sand and waste glass aggregates

Materials	Density (kg/m ³)	Fineness modulus	Maximum Size (mm)	Water absorption (%)
Natural sand	2.60	1.95	1.18	0.35
Waste glass	2.49	3.18	2.36	0.10



(b)

Fig. 5.1 Physical appearances of (a) natural sand and (b) waste glass aggregate



(a)



(b)

Fig. 5.2 SEM images of (a) natural sand and (b) waste glass aggregate



Particle size (mm)

Fig. 5.3 Grain size distributions of natural sand and glass aggregate

5.2.2 Mixing, casting and curing of test specimens

Five different mixes were used to cast the mortar specimens in the laboratory, as given in Table 5.3. In all the mixes, the mass percentages of 70% FA and 30% GGBFS were selected based on the previous studies on the fabrication of AAC under ambient curing condition [25]. The activator solution to solid binder mass ratio and aggregate to solid binder ratio were kept constant at 0.6 and 1.6, respectively. It can be seen from Table 5.3 that the aggregate to paste volumes ratio increased with increase of glass aggregate since the density of glass aggregate was lower than that of natural sand. Sodium hydroxide solution to sodium silicate solution ratio was 2. These solutions were mixed and kept in chemical storage room for 24 hours before the casting.

To cast the mortar specimens, all solid materials were placed in a Hobart mixture and mixed for about 2 to 3 minutes at a low speed. Then the premixed activator solution was added carefully through the window of the Hobart mixture and mixed again for 3 to 4 minutes using the medium speed. The freshly mixed mortar was poured into moulds of different sizes as per the test requirements. The moulds were filled in two pours of mortar and vibrated gently after the completion of each pour of mortar using a vibrating table. The cast moulds were shifted to a controlled environment at $20 \pm 3^{\circ}$ C and relative humidity of $65 \pm 10\%$. After demoulding, the samples were cured at the same temperature and humidity until the testing ages.

Mix ID			SH*		Fine aggregate		Aggregate to	
	FA	GGBFS		SS**	Sand	Waste glass	ratio	
AAMG0	481	207	138	276	1100	0	0.71	
AAMG25	481	207	138	276	825	275	0.72	
AAMG50	481	207	138	276	550	550	0.73	
AAMG75	481	207	138	276	275	825	0.74	
AAMG100	481	207	138	276	0	1100	0.75	

Table 5.3	Mix	proportions	of	alkali	activated	mortars
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*Sodium hydroxide solution (8M)

**Sodium silicate solution

5.2.3 Testing methods

To determine workability, the flow of the mortar was measured in accordance with ASTM C1437 [26] using a flow table apparatus. During the casting time, the freshly produced mortar was poured uniformly into the flow mould and then dropped the table 25 times in 15s immediately after removing of the flow mould. The difference between the diameter of flow mould base and the spread of mortar after the drops was expressed as the flow of mortar.

Fifty-mm cube mortar samples were cast to evaluate compressive strength in accordance with ASTM C109 [27] using a MATEST universal testing machine at a loading rate of 1.2 kN/sec. Since the strength of ambient-cured geopolymers increases with age [25], the compressive strength tests were conducted at 7, 14, 28 and 90 days in order to understand the strength development of the alkali activated mortars of this study.

25 mm × 25 mm × 285 mm prism specimens were used to determine drying shrinkage at 7, 14, 21, 28, 56 and 90 days by calculating the length change of the specimens as described in ASTM C157 [28]. The specimens were kept horizontally in specially designed shrinkage testing room throughout testing periods by maintaining a temperature of 20 ± 3 °C and relative humidity of 65 \pm 10%. Length of the specimen was measured using as a digital horizontal length comparator. The length of the specimen after demoulding was used as the reference length to compare the changes at each test age.

Sorptivity of the mortar sample was measured at 28 days according to ASTM C1585 [29]. At 28 days of age, disc sized test specimens of 100 mm diameter and 50 mm height were cut from cylindrical mortar samples of 100 mm diameter and 200 mm height discarding the top and bottom slices. The curved surface of the specimens were sealed using duct tape, whereas plastic sheet was loosely placed using rubber band on the top surface of the specimens to avoid evaporation from the specimens while exposed to water for sorption. The mass of two identical specimens was recorded at various time intervals up to 6 hours exposure to water as per the standard. The calculated water absorption rate of the test specimens was denoted as sorptivity of the specimens.

Rapid chloride permeability test (RCPT) was conducted to measure the chloride permeability by following test standard ASTM C1202 [31] on test specimens of 100 mm diameter and 50 mm height at 28 days using a Giatec Perma[™] testing instrument. Fig. 5.4 shows the experimental set up of RCPT. In order to conduct RCPT, the curved surface of the specimen was sealed with commercially available silicone grease and then it was vacuumed using a vacuum saturation apparatus. After that, the flat faces of each specimen were attached tightly with the testing cells. The testing cells were filled the 3% NaCl solution and 0.3N NaOH solution, respectively. The testing cells and temperature measurement sensor were connected with the main unit using cables. After 6h of testing period, the total charge (in Coulombs) passed through the specimens was recorded.



Fig. 5.4 Experimental set up of RCPT

Porosity of mortar samples of 100 mm diameter and 50 mm height was determined at 28 days as per ASTM C642 [30]. The mass of the specimen was measured successively after oven dry, boiling and suspended conditions in order to calculate porosity.

To determine the high temperature resistance, 28-days cured mortar specimens were heated to four different temperatures of 200, 400, 600 and 800°C inside a locally made electric furnace with a heating rate 5°C per minute. Several thermocouples attached with a data logger were placed inside the furnace to monitor the changes of temperature with respect to time, as shown in Fig. 5.5. The specimens were kept inside the furnace for 2 hours once the inside temperature of the furnace reached to the designated temperature. Then, the specimens were left inside the furnace for cooling down naturally and the door of the furnace was kept open during the cooling process. After cooling, the physical appearance was inspected, and the residual compressive strength was determined.

SEM and EDX examinations were done to characterize the microstructure of mortar specimens using a NEON 40EsB (ZEISS) microscopy instrument. BSD and 20 kV accelerating voltage were selected during the SEM. Small pieces of samples were cut carefully from the mortar cube specimens using a thin sized diamond saw. Then, the samples were vacuumed and coated with 10 nm carbon to avoid charges during the imaging process. After coating, the samples

were attached with sample holder using carbon tape and inserted inside the SEM chamber for examination.



Fig. 5.5 Mortar specimens inside the electric furnace

5.3 Results and discussion

5.3.1 Workability

The effect of waste glass aggregate on the workability of alkali activated FA-GGBFS mortars is shown in Fig. 5.6. It can be noticed from Fig. 5.6 that the flow of the mortars gradually increased with the increase of glass aggregate content. The use of 25% to 100% waste glass increased the flow value of mortar by 4% to 15%. The increase of flow is ascribed to the relatively high fineness modulus and smooth surface texture of glass aggregate than sand as shown in Table 5.2 and Fig. 5.3. Similar trend of flow with the use of waste glass aggregate was also reported by Lu and Poon [14]. On the other hand, Gholampour et al. [15] found that geopolymer mortars with glass aggregate showed lower flowability than that with natural sand

since the fineness modulus of glass aggregate were lower than those of natural sand. Also, Shettima et al. [32] reported that the increase of fineness modulus of fine aggregate increased workability of concrete due to the decrease of total surface area that reduced water demand. Therefore, the effect of glass aggregate on the workability of alkali activated FA-GGBFS mortars of this study showed the same trend reported in similar previous studies.



Fig. 5.6 Flow of alkali activated FA-GGBFS mortars containing different percentages of waste glass fine aggregate

5.3.2 Compressive strength

Compressive strengths of the alkali activated FA-GGBFS mortars containing different percentages of glass fine aggregate at 7, 14, 28 and 90 days are presented in Fig. 5.7. It can be observed that compressive strength of the mortars slightly decreased with the increase of glass aggregate content. The 28-day compressive strength of mortar decreased from 71 MPa to 66 MPa by the replacement of 100% sand using glass aggregate. This reason can be referred to the inadequate bond between the glass aggregate and the binder matrix due to the smoothness

of glass surface. Besides, the angularity of glass aggregate may increase the internal voids that affects the development of compressive strength negatively. Lu and Poon [14] noticed the similar trend of compressive strength for glass aggregate mortars. The authors stated that the smooth surface of glass aggregate reduced the bond between aggregate and binder. As a result, alkali activated FA-GGBFS mortars with various percentages of glass aggregate showed lower compressive strength than the mortar with natural sand only. However, as seen in Fig. 5.7, the difference of the compressive strengths for using natural sand and glass aggregate decreased with the increase of age. For example, the mortars containing 25% to 100% glass aggregate showed about 4% to 12% lower compressive strengths at 7 and 14 days than those of mortar with natural sand. However, the glass aggregate mortars exhibited only 2% to 5% lower compressive strengths than the reference mortar at 90 days for using 25% to 100% glass aggregate. The strength increase of glass aggregate mortars at later ages is attributed to the dissolution of very fine glass particles in alkaline liquid, which improved the gel network of alkali activated system by the contribution of additional silica, as discussed with microstructural evidences in the Chapter 3. Similarly, Gholampour et al. [15] found that FA geopolymer mortar containing 100% glass aggregate exhibited about 4% lower compressive strength at 7 days; however, gained slightly higher compressive strength (about 2%) at 56 days compared to FA geopolymer mortar with 100% natural sand. The authors explained that geopolymer mortars with glass aggregate gave higher compressive strength at later ages due to the partial dissolution of finer glass particles in the presence of activator solution which improved the geopolymerization process. Besides, Hajimohammadi et al. [18] reported strength increase of geopolymer mortars with glass particles after 56 days compared to that of geopolymer mortar with sand. This was due to the presence of high fraction of fine glass particles that can participate in reaction at later ages.



Fig. 5.7 Compressive strengths of alkali activated FA-GGBFS mortars containing various percentages of waste glass as fine aggregate

5.3.3 Drying Shrinkage

The effect of waste glass fine aggregate on the drying shrinkage of alkali activated FA-GGBFS mortars is shown in Fig. 5.8. It can be seen that drying shrinkage of the mortars decreased at all ages with the increase of glass aggregate content. The drying shrinkage of alkali activated mortar at 90 days gradually decreased from 1938 microstrain for 100% sand to 1620 microstrain for using 100% glass aggregate. Similarly, Hajimohammadi et al. [18] found that the use of finer glass particles as substitution of natural sand reduced the drying shrinkage of the geopolymer foams. Besides, some previous studies reported decrease of drying shrinkage

of Portland cement mortar [33-34] and concrete [35-36], with the increase of glass aggregate content. The reduction of drying shrinkage by glass aggregate is attributed to the high elastic modulus and low water absorption capacity of glass [37]. In addition, Rao [38] noticed that the increase in the size of fine aggregate decreased drying shrinkage of mortar due to the restraining effect of relatively larger size fine aggregate. As shown in Table 5.2, the maximum size of the glass aggregate was 2.36 mm, whereas the maximum size of the natural sand was 1.18 mm in this study. Consequently, the amount of larger size aggregate increased when natural sand was replaced by glass aggregate. This reduced the drying shrinkage of mortar by improved restraining action of the aggregate. Furthermore, the aggregate to paste volumes ratio has an influence on the restraining action of the aggregate [39-40]. As shown in Table 5.3, the aggregate to paste volumes ratio gradually increased with the increase of glass aggregate due to the difference between densities of sand and glass. As a result, the use of glass fine aggregate provided more restraint in ITZ, which helped to reduce the shrinkage of the glass aggregate mortars.



Fig. 5.8 Effect of waste glass fine aggregate on the drying shrinkage of alkali activated FA-GGBFS mortars

5.3.4 Sorptivity

Fig. 5.9 shows the sorptivity coefficients of alkali activated FA-GGBFS mortars with 0% to 100% glass fine aggregate. It has been noticed that sorptivity coefficients of the alkali activated FA-GGBFS mortars containing natural sand is much higher than the sorptivity coefficients of the alkali activated mortars containing 25% to 100% glass aggregate. It can be seen that sorptivity coefficients of the reference mortars decreased about 32% to 34% for using 25% to 50% glass aggregate. Sorptivity coefficient slightly increased for using 75% to 100% glass aggregate; however, these values were still lower than the values obtained for the reference mortar. For instance, mortars with 75% and 100% glass aggregate exhibited about 21% and 13% lower sorptivity coefficients as compared to the reference mortar. The lower water absorption capacity of glass aggregate is primarily responsible for the promising results of glass aggregate mortars against sorptivity. Besides, some fine glass particles might improve the pore structure by forming additional reaction products from the dissolution of reactive silica [41], as reported in the Chapter 3. However, the increase of sorptivity at higher levels of sand replacement by glass aggregate is attributed to the increase of internal voids or porosity due to the angularity of glass aggregate [42]. The results obtained in this study are consistent with the findings of Gholampour et al. [15] and De Gutierrez et al. [43], in which alkali activated FA or metakaolin (MK) mortar with 100% glass aggregate showed significantly lower water absorption than that with 100% natural sand. Furthermore, some previous studies mentioned that cement mortar containing glass aggregate showed significantly lower sorptivity values than the mortar with natural sand due to the impermeable nature of glass [44-46].



Fig. 5.9 Sorptivity coefficient of the alkali activated FA-GGBFS mortars with waste glass as fine aggregate

5.3.5 Chloride permeability

Fig. 5.10 shows the RCPT results of the alkali activated FA-GGBFS mortars containing glass aggregate. It can be noticed that the amount of total charge passed (Coulombs) through the mortar specimens decreased with the increase of glass content. The use of 25% to 100% waste glass as fine aggregate in alkali activated FA-GGBFS mortars reduced the chloride ion penetrability by about 7% to 22%. Wright et al. [35], Kou and Poon [47], and Wang [48] reported similar trends for using waste glass fine aggregate in Portland cement concrete. The water absorption of glass aggregate is very little or negligible as well as the surface of the glass is mainly smooth when compared to sand (Table 5.2 and Fig. 5.2), therefore, glass aggregate acted as an impermeable membrane to moisture and ions into the matrix [35]. As a result, the use of glass as aggregate exhibited positive influence against chloride permeability of mortar.

In addition, it can be noted that the amount of total charge passed through the specimens of the current study is comparatively higher than the values reported in some previous studies [47-48] for OPC based specimens. The variation of the results can be referred to the higher pore solution conductivity of alkali activated composites as compared to that of OPC based binder system [49-50]. In the present study, chloride ion permeability of the alkali activated FA-GGBFS mortars with 100% natural sand can be classified as "high", whereas chloride ion permeability of mortars with 25% to 100% glass aggregate can be classified as "moderate" as per ASTM C1202 [31].



Fig. 5.10 Effect of waste glass fine aggregate on the chloride permeability of alkali activated FA-GGBFS mortars

5.3.6 Porosity

The effects of using 0% to 100% waste glass as fine aggregate on the porosity of the alkali activated FA-GGBFS mortars are presented in Fig. 5.11. It can be seen that porosity of the mortars increased with the increase of glass aggregate percentage. The use of 25% to 100% glass aggregate as a replacement of natural sand increased porosity of the mortars by 1.5% to 13%. This is similar to the findings of Bisht and Ramana [42] and Ouldkhaoua et al. [51] reporting that porosity of conventional cementitious systems gradually increased with the increase of glass aggregate content. From Fig. 5.10, it is also noticed that the increase in porosity of the mortars is more prominent when 75% and 100% sand was replaced by glass aggregate. This observation show agreement with the decreasing trend of compressive strength results presented earlier. The poor bond strength between the smooth surface of glass aggregate and the paste resulted in an increase of porous nature in the ITZ of the glass aggregate mortars. Besides, glass aggregates are mainly angular and irregular in shape, which increased the amount of entrapped air [36]. In addition, the microcracking caused by oven-drying during porosity testing may induce additional porosity in the glass aggregate mortars. Furthermore, the very high replacement of sand by glass aggregates reduced particle packing density which led to the increase in porosity since glass aggregate was coarser than the natural sand [46]. Though porosity increased with the increase of glass aggregate, the sorptivity and RCPT results presented in sections 5.3.4 and 5.3.5 appear to have weak correlations with porosity. Generally, permeability properties of cementitious matrix such as sorptivity and chloride permeability are varies with porosity as well as interconnection among the pores [52-53]. The use of waste glass as aggregate increased the volume of pores in the matrix due to its smooth surface texture and irregular shape [42, 51]. However, the interconnectivity among these pores is less than that of the matrix using sand aggregate [18]. Besides, the water absorption of glass particles is less than sand, as shown in Table 5.2. These are considered as the possible reasons for weak correlations of porosity with the sorptivity and RCPT results of mortars using glass aggregate.



Fig. 5.11 Effect of waste glass as fine aggregate on the porosity of alkali activated FA-GGBFS mortars

5.3.7 Resistance to high temperature exposure

5.3.7.1 Residual strength

The residual compressive strengths of the alkali activated FA-GGBFS mortars containing 0% to 100% glass aggregate after exposure to 200°C to 800°C temperatures are presented in Fig. 5.12. Fig. 5.12 indicates that all the mortars gained some strength after exposure to 200°C and the compressive strength of the mortars varied from 82.8 MPa to 72.8 MPa for using 0% to 100% glass aggregate. The additional reaction of the binder particles at this temperature regime may have triggered the increase of strength. Similarly, Sarker et al. [54] reported that geopolymer concrete gained additional compressive strength than the initial compressive strength after exposed to 200°C as a result of further geopolymerization. Besides, Bernal et al. [55] noticed that alkali activated GGBFS pastes showed slight increase in compressive strength

as compared to its initial strength when exposed at 200°C. It has been also noticed from Fig. 5.12 that the residual compressive strength of the mortars after exposed to 200°C and 400°C followed the similar trend for using waste glass that was observed for compressive strength of the mortars tested after ambient curing. However, glass aggregate mortars showed higher compressive strength than the reference mortar after exposed to 600°C and 800°C. For instance, the compressive strength of the mortars containing 25% to 100% glass aggregate were 23.8, 24.5, 25.6 and 25.6 MPa, respectively, whereas the compressive strength of the mortar containing 100% natural sand was 23.7 MPa after 800°C exposure. This findings show good agreement with the results reported by Lu and Poon [14]; Ouldkhaoua et al. [51] and Lu et al. [56]. Lu and Poon [14] mentioned that the thermal expansion coefficient of sand is much higher than the glass. Consequently, the use of glass as fine aggregate can compensate the damages of the aggregate-binder interface zone due to the less expansion under elevated temperature at 800°C. Besides, Yang et al. [57] described that concrete mixtures containing glass aggregate showed higher temperature resistance than the control concrete due to the following reasons: (i) softened glass aggregate improved the microstructure by pore filling effect after exposed to high temperature and (ii) surface of the glass aggregate softened at 800°C improved the bond strength of the aggregate paste interface. Furthermore, the reason behind the satisfactory performance of glass aggregate against high temperature will be discussed elaborately by observing visual appearances and microstructures after exposed to high temperatures in the later sections.



Fig. 5.12 Residual compressive strengths of the alkali activated FA-GGBFS mortars containing glass fine aggregate after exposure to elevated temperatures

5.3.7.2 Visual observations

The physical appearances of the alkali activated FA-GGBFS mortars containing 0%, 50% and 100% glass aggregate after 200°C to 800°C exposures are presented in Figs. 5.13, 5.14 and 5.15, respectively. It has been noticed that the mortar specimens did not experience any significant damages or changes on the surfaces except change in colour after 200°C to 600°C exposures. It can also be observed that the presence of pores on the surface increased in the temperature ranges between 200°C to 600°C. This may due to the release of physically bound water from the specimens at this temperature regime. In addition, it is worth to mention none of mortar specimens showed any spalling or physical damage at any stage of the experiment. After exposure at 800°C, AAMG0 mortar specimens experienced some severe longitudinal cracks along with transverse or pattern type cracks on the surface as presented in Fig. 5.13 (d). As seen in Figs. 5.14 (d) and 5.15 (d), there is no sign of any visible longitudinal cracks was in AAMG50 and AAMG100 mortar specimens after exposed at 800°C. On the other hand, AAMG0 mortar specimens experienced some severe longitudinal cracks along with transverse or pattern type cracks on the surface as gresented in Fig. 5.13 (d).

or pattern type cracks on the surface after exposure at 800°C as presented in Fig. 5.13 (d). Besides, these mortars only experienced pattern type cracks which was distributed throughout the whole surface as presented in Figs. 5.14 (d) and 5.15 (d). This is expected since the thermal expansion coefficients of sand is almost twice of that of the glass [14]. As a result, the volume of mortar containing 100% sand (AAMG0) increased substantially after 800°C exposure that led to formation of both longitudinal and transverse cracks on the surfaces. Therefore, apparently, it can be said that the formation of longitudinal cracks is primarily responsible for the lower residual strength of AAMG0 mortar specimens at elevated temperatures.



Fig. 5.13 Appearances of AAMG0 samples after exposure to elevated temperatures



Fig. 5.14 Appearances of AAMG50 samples after exposed to elevated temperatures



Fig. 5.15 Appearances of AAMG100 samples after exposed to elevated temperatures

5.3.8 SEM and EDX examinations

The SEM images of AAMG0, AAMG50 and AAMG100 mortar specimens cured at ambient temperature are shown in Figs. 5.16, 5.17 and 5.18. Referring to Fig. 5.16, it can be seen that the microstructure of AAMG0 mortars is consisted with a strong and uniform bonding between the paste matrix and sand aggregate at the interfacial transition zone (ITZ). On the other hand, a weak bond is observed between the paste matrix and glass aggregate at the ITZ of the AAMG50 and AAMG100 mortars as shown in Figs. 5.17 and 5.18. This weak bond or

connection at the ITZ of glass aggregate mortars is attributed to the smooth surface texture of glass. This is in good agreement with the microstructural observations reported by Liu et al. [46], where the authors reported the effect of glass aggregate on the microstructure of cementbased concrete. Besides, it has been noticed that the presence of internal voids and microcracks in AAMG50 and AAMG100 mortars (Fig. 5.17 and Fig. 5.18) is significantly higher than those of AAMG0 mortar (Fig. 16). This observation supports the porosity results of mortars as discussed briefly in section 5.3.6. It is worth mentioning that microcracks can be formed during the preparation of the samples. However, it can be seen from Figs. 5.17 and 5.18 that some microcracks might be started from the sharp edge of the glass aggregate which propagated through the microstructure later on. From above discussion, it is obvious that the addition of glass as substitution of natural sand reduced compressive strength of the mortar due to the following reasons: (i) weak bond at the ITZ of the glass aggregate and (ii) irregular and angular glass with sharp edge introduced microcracks or internal gap in the microstructure.



Fig. 5.16 SEM image of ambient cured AAMG0 mortar specimen



Fig. 5.17 SEM image of ambient cured AAMG50 mortar specimen



Fig. 5.18 SEM image of ambient cured AAMG100 mortar specimen

In addition, the SEM images of AAMG0, AAMG50 and AAMG100 mortar specimens after 800°C exposure are presented in Figs. 5.19, 5.20 and 5.21 to understand the effect of glass aggregate on high temperature exposures. As seen in Fig. 5.19, the ITZ of AAMG0 mortar specimen is severely damaged after exposure at 800°C. This may be due to the abnormal expansion of the sand aggregate at 800°C which created stress at the ITZ. As a result, the bonding between sand and paste at the ITZ has weakened. This observation also validates the visual appearance of AAMG0 mortar after 800°C exposure as shown in Fig. 5.13 (d). On the other hand, it can be seen in Figs. 5.20 and 5.21 that partially softened glass formed a good connection with the paste matrix at the ITZ of the AAMG50 and AAMG100 mortar specimens after 800°C exposure. Besides, it is also noticed that the ITZ of the AAMG100 mortar specimen is relatively more homogeneous and less porous as compared to the microstructure of AAMG0 mortar specimen (Figs. 5.19 and 5.21).



Fig. 5.19 SEM image of AAMG0 mortar specimen after 800°C exposure



Fig. 5.20 SEM image of AAMG50 mortar specimen after 800°C exposure



Fig. 5.21 SEM image of AAMG100 mortar specimen after 800°C exposure

Furthermore, the elements obtained from EDX analysis of AAMG0 (points A, B, C) and AAMG100 (points D, E, F) after 800°C exposure are given in Table 5.4. It can be seen that the Si content was generally higher in the paste of AAMG100 than in AAMG0. This is similar to the observation of Zhang et al. [58] reporting that the paste matrix of glass aggregate mortar exhibited high intensity of silicon in EDX analysis after 800°C exposure. The higher Si content is considered indicative of the partial softening of glass particles at 600°C and higher temperatures that may have contributed to improve the bonding and give slightly higher residual strength. Similar residual strengths were also reported by Lu et al. [14] and Yang et al. [57]. Therefore, the mortars with glass aggregate showed higher residual compressive strengths than the mortars containing natural sand when subjected to elevated temperatures as discussed in the earlier section.

Mix ID	Spectrum	0	Si	Ca	Al	Na
		(Wt %)				
AAMG0	А	44.7	23.0	11.6	6.9	7.8
	В	46.9	23.3	8.9	7.1	7.6
	С	56.2	21.9	7.8	6.7	5.4
AAMG100	D	39.4	37.1	8.8	5.2	5.5
	Е	44.8	38.9	6.4	3.3	4.4
	F	41.3	37.5	10.9	6.8	1.8

 Table 5.4 EDX analysis results

5.4 Summary

The experimental results presented in this chapter are summarized below:

- The use of 25% to 100% glass aggregate increased the flow of alkali activated FA-GGBFS mortars by 4% to 15%. The increase of workability with the increase of glass aggregate is attributed to the higher fineness modulus, smooth surface texture and lower water absorption capacity of glass than sand.
- 2. Compressive strength of alkali activated FA-GGBFS mortars containing 0% to 100% glass fine aggregate continued to increase up to 90 days of age. The compressive strength at 28 days decreased from 71 MPa to 66 MPa for replacing 100% sand by glass aggregate. The slight decrease of strength is attributed to the smooth surface texture that deceased the bond between aggregate and binder as well as increased the presence of pores in the bulk matrix.
- Drying shrinkage of alkali activated FA-GGBFS mortars decreased significantly for using 25% to 100% glass aggregate due to the lower water absorption and higher elastic modulus of glass aggregate.
- 4. Sorptivity coefficient of alkali activated FA-GGBFS mortars significantly decreased for using 25% and 50% glass aggregate, and then increased for 75% and 100% glass aggregate. The lower water absorption capacity of glass aggregate is primarily responsible for the improvement of sorptivity when 25% or 50% sand is replaced using glass aggregate. However, the increase of sorptivity at higher levels of sand replacement (75% and 100%) by glass aggregate is attributed to the increase of internal voids or porosity due to the angularity of glass aggregate. Besides, the mortars with 25% to 100% glass aggregate exhibited about 1.5% to 13% higher porosity as compared
to the mortar without glass aggregate. The increase of porosity is considered to be contributed by the more angular shape of glass particles as compared to natural sand.

- 5. Chloride permeability of alkali activated FA-GGBFS mortars significantly decreased for using 25% to 100% glass aggregate since glass aggregate acted as an impermeable membrane to moisture and ions into the matrix.
- 6. The residual strength of the alkali activated FA-GGBFS mortars showed slightly decreasing trend with the increase of glass aggregate when exposed at 200°C and 400°C. However, a slightly increasing trend of residual strength was noticed with the increase of glass aggregate after 600°C and 800°C exposures. A possible reason for the variation may be attributed to the difference in thermal expansions of natural sand and glass aggregates.
- 7. SEM images revealed that the microstructure of mortar using glass aggregate was associated with higher amount of internal voids and weaker adhesion at the ITZ, which is primarily responsible for the lower initial compressive strength of these mortars. However, partially softened glass introduced better bond with the paste matrix at the ITZ at the exposures of 600°C to 800°C. As a result, alkali activated FA-GGBFS mortars containing waste glass aggregate showed satisfactory results against high temperature exposures.

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CHAPTER 6

PERFORMANCE OF ALKALI ACTIVATED MORTARS CONTAINING WASTE GLASS FINE AGGREGATE AGAINST ACID EXPOSURES

The contents presented in this chapter is submitted to the Advances in Cement Research Journal for possible publication, 2021. (Under review).

This chapter investigates the influence of waste glass cullet as fine aggregate in alkali activated FA-GGBFS mortars exposed to H_2SO_4 and HCl acid solutions for one year. The changes in physical appearance, surface alkalinity, mass, compressive strength and microstructure of the hardened samples after immersion in acid solutions were studied.

6.1 Overview

Since natural sand is the primary source of fine aggregate for construction purposes, excessive sand mining occurs in many countries to meet the high demand of sand. This has harmful effects to the aquatic life and equilibrium of hydraulic structures [1]. Consequently, the reuse of waste glass cullet as a fine aggregate in AAC has been considered as an effective approach to reduce the negative environmental issues related to the consumption of natural sand as a construction material [2]. Some previous works showed that the use of waste glass cullet as a partial or entire substitution of natural sand would provide several advantages to the fresh and hardened properties of AAC [3-4]. For instance, Lu and Poon [3] evaluated the efficacy of waste glass cullet as a fine aggregate on the workability, compressive strength and fire resistance performance of AAC. Similarly, Gholampour et al. [4] studied some important durability properties such as water absorption, drying shrinkage and alkali silica reaction of AAC containing waste glass fine aggregate. Recently, Saccani et al. [5] evaluated the ASR, sulphate resistance and resistance to freeze-thaw cycles of AAC associated with various types of waste glass cullet. Moreover, the findings of the previous studies suggest that the use of waste glass as fine aggregate offers equivalent or even better hardened properties to those using natural fine aggregate. However, studies related to the stability of AAC containing glass fine aggregate under aggressive exposures such as acid resistance are scarce in the literatures. Therefore, it is essential to evaluate the efficacy of waste glass cullet as a fine aggregate on the durability of AAC since concrete structures built in marine, mining and industrial areas frequently experience aggressive acidic surroundings during their service life [6-7]. Considering the above perspectives, this chapter evaluates the influence of waste glass cullet as a fine aggregate on the acid resistance of FA and GGBFS blended AAC. Natural sand was

substituted by waste glass cullet at various percentages and the hardened samples were exposed to both H₂SO₄ and HCl solutions for one year. The effects of waste glass fine aggregate are evaluated in terms of the changes in visual appearance, surface alkalinity, mass, mechanical and microstructural properties of the samples.

6.2 Experimental work

6.2.1 Materials and preparation of test specimens

In this study, FA and GGBFS were used as aluminosilicate precursors to prepare alkaliactivated mortars. Mixture of commercial sodium silicate (silica modulus = 2) solution and sodium hydroxide solution (8 molar concentration) was used as an alkaline solution to activate the aluminosilicate materials. Waste glass cullet and natural silica sand were used as the fine aggregates. The physical and chemical properties of the materials used in this study are briefly discussed in the Chapter 5. In this study, the combination of 70% FA and 30% GGBFS (by mass) were selected as aluminosilicate materials to fabricate alkali activated mortars. Five different mixtures were studied with a varying percentage of natural sand and glass aggregates as presented in Table 6.1. The solution to powder mass ratio and aggregate to solid binder ratio were fixed at 0.6 and 1.6, respectively. The ratio of sodium hydroxide solution to sodium silicate solution was 2 and the solution was prepared 24 hours before it was used.

Table 6.1 Mix proportions of alkali activated mortars

		Constituents (kg/m ³)					
Mix ID					Fine		
	FA	GGBFS	SH*	SS**	Sand	Waste glass	Waste glass (%)
AAMG0	481	207	138	276	1100	0	0
AAMG25	481	207	138	276	825	275	25
AAMG50	481	207	138	276	550	550	50
AAMG75	481	207	138	276	275	825	75
AAMG100	481	207	138	276	0	1100	100

*Sodium hydroxide solution (8M)

******Sodium silicate solution

At the beginning of the mixing, all solid materials were dry mixed about 3 minutes using a laboratory mixture machine. Following this, the alkaline solution was added and mixed for additional 4 minutes. The fresh mortar was placed in 50 mm cube moulds to cast the test specimens. Afterward, it was stored at $20 \pm 3^{\circ}$ C with a relative humidity of $65 \pm 10\%$ and cured until tested.

6.2.2 Acid resistance test

After 28 days of ambient curing, the initial mass, compressive strength and porosity of the mortar specimens were recorded. Compressive strength was evaluated as per ASTM C109 [8] and porosity was measured according to ASTM C642 [9] test standards. The details of these tests were described in the Chapter 5. Following that, the specimens were immersed in 3% H₂SO₄ and 3% HCl solutions separately and stored in ambient temperature throughout the designated exposure periods. The acid solutions were refreshed in every month with caution so that no damage occurred to the samples. The mass measurement and compressive strength test were performed at 30, 90, 180, 270, and 365 days of exposure in the acid solutions. Besides, the mortar samples were visually inspected regularly to identify any external cracks, spalling or deterioration due to acid attack. The surface alkalinity of the acid exposed samples were determined by spraying 1% phenolphthalein solution on the cross-section of the samples at the end of acid exposure tests, as described in some previous studies [10-11]. Careful handling and precise mass measurement were performed in order to avoid any damage of the specimens, especially at the later ages. Furthermore, in order to evaluate the influence of acid solution on the aggregate's morphology and microstructure, the natural sand and waste glass cullet were also immersed in 3% H₂SO₄ and 3% HCl acid solutions throughout the designated exposure period. The microstructures of acid exposed samples were examined by SEM technique with a NEON 40EsB (ZEISS) microscope. The SEM images were captured using BSD mode. A thin section of mortar was cut using a diamond saw. It was then vacuumed adequately after drying and coated with 10 nm carbon. Then, a sample holder was used to properly set the specimen and place into the SEM chamber for examination.

6.3 Results and discussion

6.3.1 Hardened properties of the unexposed samples

The compressive strength and porosity results of the unexposed mortars cured for 28 days are presented in Fig. 6.1 and Table 6.2, respectively. It can be seen that the AAC with 100% silica sand showed the highest strength of 71.1 MPa and the lowest porosity of 15.7%. Fig. 6.1 shows that compressive strength is decreased and porosity is increased with an increase of waste glass aggregate in the mixture. Table 6.2 shows that the compressive strength decreased by 7.59% when 100% sand was substituted by waste glass fine aggregate. At this replacement level, the porosity is increased by 29.94%. It can be seen that there were relatively small changes in strength and porosity for the sand replacement up to 50%. However, as shown in Table 6.2, the porosity increased at higher rates when 75% and 100% sand was substituted by glass fine aggregate though compressive strength decreased at a smaller rate. The low decrease of strength for higher glass aggregate contents is attributed to the interlocking characteristic of angular glass particles that could enhance compressive strength.



Fig. 6.1 Relationship between the compressive strength and porosity of mortars tested at 28

Mix ID	Compressive strength (MPa)	% Decrease in strength compared to 100% sand	Porosity (%)	% Increase in porosity compared to 100% sand
AAMG0*	71.1	0	15.7	0
AAMG25	69.5	2.25	15.9	1.27
AAMG50	68.3	3.94	16.6	5.73
AAMG75	67.6	4.92	18.0	14.65
AAMG100	65.7	7.59	20.4	29.94

Table 6.2 Compressive strength and porosity of mortars tested at 28 days (unexposed)

*Indicates the control mixture with 100% silica sand

The reduction of compressive strength with incorporation of glass aggregate can be referred to weaker bond of the smooth surface textured glass aggregate to the binder matrix [3, 12]. Moreover, the increase of crushed glass particles can also create internal voids due to their angular geometry which may increase the susceptibility of crack propagation, and thus reducing compressive strength. A number of studies reported that the porosity of increased with increased amount of glass aggregate [13-14]. The increase of angular and irregular crushed glass particles can entrap more air and increase the porosity [15-16]. Furthermore, the particle packing can be significantly affected by the use of a very high percentage of glass aggregate resulting in an increased porosity [15].

6.3.2 Evaluation of acid resistance

6.3.2.1 Bulk and microscopic changes of aggregates

Figs. 6.2 and 6.3 present the bulk and SEM images of sand and glass aggregate before and after immersion in the acid solutions. It can be seen that the original colours of sand and glass aggregates entirely changed when immersed in 3% H₂SO₄ for 1 year. However, no obvious physical change was noticed in these aggregates after exposure to 3% HCl for 1 year. The SEM images reveal that the surface of the sand and glass aggregate slightly decayed after H₂SO₄ exposure; however, there is no evidence of acute surface degradation or cracking. On the other hand, after HCl exposure, the morphology of these aggregates showed similar texture to that of the original samples.









Gu et al. [17] found that limestone aggregate severely deteriorated after 1% H₂SO₄ acid exposure for 14 days. The authors claimed that limestone aggregate consisted of usually 43% to 46% CaO, therefore, these aggregates promoted the formation of gypsum crystals when

exposed in acidic environment. It is well known that the primary constituent of silica sand and glass aggregate is SiO_2 , which commonly act as inert in acidic media [18]. Consequently, these aggregates showed low or negligible degradation when exposed in acid solutions. Overall, it is evident that sand or glass aggregate are less susceptible to acid solutions; however, the significant differences in physical and morphological characteristics between the sand or glass aggregates may have influenced the acid resistance performance significantly that are further discussed in the following sections.

6.3.2.2 Change in physical appearance and surface alkalinity of mortar specimens

Figs. 6.4 and 6.5 show the physical appearance of mortar samples after 1 year exposure in H₂SO₄ and HCl acid solutions. As shown in Fig. 6.4, all samples experienced significant deterioration at the edges along with deposition of yellowish white substances on the surface after H₂SO₄ exposure. Some visible cracks were found at the edges of the samples. It can be noticed that the presence of such cracks were more intense in the high volume glass aggregate mortar samples such as AAMG75 and AAMG100. However, the samples submerged in HCl solution did not exhibit signs of significant physical damage except some minor changes in the texture of the surface due to the deterioration, as observed in Fig. 6.5. However, this surface deterioration is also more prominent in the mortar samples using glass aggregate.



Fig. 6.4 Physical appearance of the mortar samples after one year in 3% H₂SO₄ exposure



Fig. 6.5 Physical appearance of the mortar samples after one year in 3% HCl exposure The apparent degradation due to acid exposure as monitored by applying 1% phenolphthalein solution on the cross-section of the acid exposed samples, are presented in Fig. 6.6 and 6.7.



Fig. 6.6 Cross sections of the H₂SO₄ exposed samples sprayed with phenolphthalein solution (colourless edges-Degraded area; coloured core-Un-degraded area)





Apparently, all samples exhibited a pink colour core and a colourless outer layer. However, a significant variation related to perimeter of coloured core among the samples can be noticed.

The perimeter of the coloured core decreased with the increase of glass aggregate content. This phenomenon reveals that the mortar specimens using glass aggregate allowed the transport of significant amounts of acid solutions due to their high porosity (Table 6.2) that caused severe degradation. Consequently, the mortar samples with glass aggregate showed relatively lower physical resistance against acid attacks than that of the mortar with sand.

6.3.2.3 Change in mass

The change of mass of the mortar samples after immersion in 3% H₂SO₄ and 3% HCl acid solutions at different testing periods until 1-year are shown in Figs. 6.8 and 6.9, respectively. It reveals that the mortar specimens attained some mass after 28 days of exposures in both of the acid solutions. This is similar to the observations reported by Kwasny et al. [35] and Valencia- Saavedra et al. [36] that AAC gained a slight mass initially after exposure to acid solutions.



Fig. 6.8 Change of mass of the mortars exposed to 3% H₂SO₄ up to one year



Fig. 6.9 Change of mass of the mortars exposed to 3% HCl up to one year

When the samples were submerged in acid solution, it penetrated through the pores of the samples and formed expansive salts. These salts deposited into the pores of samples without causing any stress at the initial stage [19-20], which increased the mass of the samples. Afterwards, a linearly decreasing trend of mass loss of mortars was noticed starting at 28 days and continued until 365 days of exposure to both of the acidic solutions. The results revealed a higher mass reduction with increased waste glass aggregates in both the acidic environments. For instance, the mass loss of mortars with 25% to 100% glass aggregate was 14.1% to 16.8% after 1 year of immersion in H_2SO_4 solution while it varied from 10.3% to 11.90% in case of exposure in HCl solution. The gradual increase in mass loss over the exposure period can be explained by the chemical interaction of the mortar's microstructure and the acidic species that penetrated into the matrix. The effect of this reaction is manifested by the surface scaling, cracking along the edges and spalling of mortar specimens as presented in Fig. 6.4 and 6.5. As

seen in Figs. 6.8 and 6.9, a higher mass loss was recorded in the H_2SO_4 acid exposed samples than the HCl acid exposed samples at all testing ages. This is expected since the mortars exposed to H_2SO_4 acid solution experienced more physical damages than those exposed in HCl acid solution. Besides, the surface deterioration and chemical reaction between the acids and the aluminosilicate products can alter the microstructure and lead to mass loss. During the H_2SO_4 acid exposure, the H⁺ ions from H_2SO_4 weakened the framework of hydrated phases and the HSO₄⁻ ions triggered the formation of crystalline gypsum by reacting with the available Ca^{2+} released from the precursor materials [21-22]. Gypsum formation causes mass and strength loss as reported in different studies [23] which can be a potential reason for higher mass loss of AAC specimens immersed in H₂SO₄ solution.

6.3.2.4 Change in compressive strength

The change of compressive strength of the mortars exposed to 3% H₂SO₄ and 3% HCl acid solutions for different exposure periods until 1-year are shown in Figs. 6.10 and 6.11, respectively. It shows that the reduction in strength increased with an increase in glass aggregate content and exposure period irrespective of acid solutions. Similarly, Parthiban and Thirugnanasambandam [24] noticed that AAC mortars with glass aggregate experienced comparatively a higher strength reduction than those made with natural fine aggregate when exposed to HCl acid solution. In addition, it can be observed that the reduction in strength of the mortar specimens was higher when immersed in H₂SO₄ acid solution than those immersed in HCl acid solution. The intrusion of acid into the matrix is a slow process at the initial stage which is mainly encountered by the geopolymerization process. This effect might have led to a negligible strength reduction at 28 days, as shown in Figs. 6.10 and 6.11. Afterwards, a gradual decrease in strength was observed for all the mortar mixtures with their corresponding increased waste glass aggregate content. In H₂SO₄ exposure, the strength loss of mortars with 25% to 100% glass aggregate was 36.9 % to 52.1% while it was 24.6% to 38.6 % for exposure in HCl solution. It is worth to mention that the strength reduction of AAMG50 was 10.9% and 7.3%, respectively, in H₂SO₄ and HCl solutions compared to the control mixture (AAGM0). This indicates a superior performance of waste glass aggregates combating acid attack when replacing 50% natural sand. Studies have shown that the strength loss in acidic medium occurs due to the disintegration of the oxy-aluminium bridge (Si-O-Al) in the gel, which mainly works as the skeleton of the aluminosilicate matrix [25-26]. The penetration of acids and generation of unstable products increased with the exposure time as manifested by the surface deterioration shown in Figs. 6.4 and 6.5. When a certain amount of sand is replaced using the

same amount of glass aggregate that eventually leads to reduce the strength as shown in Table 6.2. This is due to the weak bond at the ITZ provided by the smooth surface textured glass aggregates [12, 16]. When mortars containing glass aggregate were submerged in acid solution, the intruded acids reacted with the aluminosilicate gel and further weakened the bond in interfacial transition zone (ITZ). These hypotheses are discussed briefly through a microstructural examination in the following section.



Fig. 6.10 Change of compressive strength of the mortars exposed to 3% H₂SO₄ up to one year



Fig. 6.11 Change of compressive strength of the mortars exposed to 3% HCl up to one year

6.3.2.5 Microstructural examinations

The microstructural changes of the reported mortars after exposure to H₂SO₄ and HCl solutions as investigated by SEM analysis are presented in Figs. 6.12 and 6.13.



(a) AAMG0



(b) AAMG50



(c) AAMG100

Fig. 6.12 SEM images of mortar samples after exposed to H₂SO₄ acid solution

It can be noticed that the microstructure of the mortars exposed to acid solutions consists of a significant amount of cracks, voids and un-hydrated or unreacted particles. Besides delamination between the paste and aggregates can also be observed at the interfacial transition zone (ITZ). This phenomenon is due to the collapse of aluminosilicate network in acidic medium which led to disintegrate the bonding between paste matrix and aggregate [7]. Since the amount of the binder component was same in all the mixes, the overall microstructural changes after the acid exposure can be referred to the replacement level of glass aggregate content in these mixtures. As seen in Fig. 6.12 (a-c), the bonding at the ITZ is seen to be more detached in AAMG100 and AAMG50 samples as compared to the AAM0 sample after exposure to H₂SO₄ acid solution. It has been reported that glass aggregate usually provides loose bonding at the ITZ partly because of its smooth texture [16]. Consequently, when the mortar was submerged glass aggregate submerged in acid solution, the glass aggregate shows a weaker bond to the matrix at ITZ. As a result, the mortars with glass aggregate showed a relatively higher reduction in strength after exposure to H₂SO₄ acid solution, as reported in the earlier section. Similarly, as observed in Fig. 6.13 (a-c), the microstructural damages such as cracks and disintegrated ITZ as well as the presence of unreacted particles are comparatively

higher in AAMG100 and AAMG50 samples than in the AAMG0 sample when exposed in HCl acid solution. Meanwhile, it is evident that the microstructural damages are less severe in HCl exposed samples as compared to the H₂SO₄ exposed samples. The generation of expansive gypsum crystals in the H₂SO₄ exposed samples is primarily responsible for the greater physical and microstructural damages as manifested in several previous studies [21-23]. These observations agree the microscopic changes of aggregates and strength loss results as reported in the previous sections.



(a) AAMG0



(b) AAMG50



(c) AAMG100

Fig. 6.13 SEM images of mortar samples after exposed to HCl acid solution

6.4 Summary

The experimental results presented in this chapter are summarized below:

1. The alkali activated mortar specimens incorporating 25% to 100% glass aggregate experienced more physical damage as well as degraded area when immersed in the acid solutions compared to the mortar specimens with natural sand aggregate. This is because of the high porosity of the glass aggregate mixed mortars which allowed the transport of significant amounts of acid solutions and caused severe degradation.

2. The mass and strength reduction due to H_2SO_4 and HCl acid attacks proportionally increased with the glass aggregate content and exposure period. After 1 year of exposure in 3% H_2SO_4 acid solution, the mortar containing 25% to 100% glass aggregate showed 14.8% to 16.8% mass loss and 36.9% to 52.1% strength loss, whereas the mass and strength losses of the reference mortar were 14.1% and 31.2%, respectively. Similar trend was observed in case of 1-year exposure in HCl acid. Overall, the reduction in mass and strength of mortars with 25% to 50% glass aggregate was comparable to those of the reference mortars. The addition of glass aggregate eventually leads to reduce the strength due to the weak bond at the ITZ as provided by the smooth surface textured glass aggregate. When mortars containing glass aggregate were submerged in acid solution, the intruded acids reacted with the aluminosilicate gel and further weakened the bond in ITZ. Consequently, strength loss of the mortars due to the acid attack increased with the increase of glass aggregate content.

3. The SEM images revealed that mortars with varying glass aggregate content experienced relatively higher microstructural deterioration than that of mortar with sand aggregate under both the acidic environments. This is because of the geometry, surface and shape characteristics of the glass aggregate which can lead to weaker bonding at the ITZ.

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CHAPTER 7

ASSESSMENT OF ALKALI SILICA REACTION OF WASTE GLASS FINE AGGREGATE BY ACCELERATED MORTAR BAR TEST

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This chapter investigates the potential ASR of waste glass cullet in alkali activated mortar, using AMBT method, in comparison to that in OPC mortar. In this regard, the AMBT expansions, mass change, compressive strength, visual appearances and microstructures of the mortar bar specimens were studied.

7.1 Overview

The extensive use of natural sand in construction has become one of the major environmental concern in many parts of the world. Consequently, research on alternative fine aggregates has drawn keen interest of the scientific community in order to meet the increasing demand for construction of infrastructures and residential buildings [1-2]. Past research shows that crushed waste glass could be a potential candidate for use as fine aggregate in cement composites [3-5]. However, the main concern of using crushed glass as fine aggregate with Portland cement binder is the potential ASR, which may cause excessive expansion and cracks [6-7]. Numerous studies found that the use of waste glass as fine aggregate in conventional cement concrete and mortar introduced severe cracks on the surface due to expansion by ASR [8-10]. However, the ASR susceptibility of reactive aggregates with alkali activated binders was reported as relatively low [11-12]. For instance, Fernandez-Jimenez and Puertas [13] and Puertas et al. [14] found that mortar specimens containing reactive aggregate (Opal aggregate) showed higher expansion with OPC than with alkali activated GGBFS. It was stated that the presence of portlandite in OPC mortar promoted the ASR by providing Ca²⁺ into the solution, whereas most of the free alkalis were consumed in the reaction products of alkali activated GGBFS. Similarly, Shi et al. [15] reported that alkali activated GGBFS binder reduced ASR expansion by lowering the alkalinity of pore solution. Besides, Fernandez-Jimenez et al. [16] and Kupwade-Patil and Allouche [17] found that both reactive (quartz and sandstone) and nonreactive (siliceous limestone) aggregates showed less expansion in alkali activated FA mortar as compared to the OPC based mortar in AMBT. The low expansion was attributed to the low calcium content of FA. In contrast, Bakharev et al. [18] reported from concrete prism test results that ASR of a reactive coarse aggregate was higher in alkali activated GGBFS

concrete than in OPC concrete. The type and composition of the reactive aggregate were not given. However, the authors explained that the pore solution of alkali activated GGBFS concrete had high alkali content which attacked the reactive aggregates and formed deleterious ASR gel. Xie et al. [19] found that the ASR expansion of glass aggregate (50-100%) in sodium silicate activated FA mortar was within the limit specified by testing standards. The authors explained that reactive glass aggregates formed low viscosity ASR type gel in alkali activated FA binder due to the low calcium content of FA. It was argued that this low-viscosity gel could easily enter into the surrounding pores instead of creating internal stress. However, Garcia-Lodeiro et al. [20] reported that alkali activated FA mortar specimens containing 100% reactive opal aggregate showed severe deterioration in AMBT. The authors revealed that some expansive zeolitic crystalline compounds were formed in alkali activated FA mortar containing reactive opal aggregates under AMBT condition. In addition, Pouhet and Cyr [21] investigated the ASR mechanism of six different types of aggregate (marble sand, siliceous sand, quartzite sand, sedimentary siliceous limestone sand, alluvial sand and crushed glass sand) in alkali activated metakaolin mortars. The authors explained that metakaolin based alkali activated binder showed satisfactory performance against ASR by the (i) reduction of pH of the pore solution, (ii) formation of low-calcium hydration product and (iii) distribution of gel thorough the porous geopolymeric network. Angulo-Ramirez et al. [22] reported that reactive basaltic aggregate mixed with volcanic glass in alkali-activated GGBFS blended with 20% OPC showed less ASR expansion than the OPC mortar. The authors argued that reactive silica present in the GGBFS and aggregate contributed to produce greater amount of reaction product in the presence of alkaline activator that increased the adhesion between paste and aggregate instead of producing expansive products.

It can be seen from the above discussion that generally, alkali activated GGBFS and conventional OPC binders contained calcium rich reaction products, whereas alkali activated binders using FA produced low calcium reaction products. Different types of reactive aggregates, different aluminosilicate sources and different activators were used in the previous studies. This chapter focused on the ASR susceptibility of reactive waste glass aggregate in alkali activated neat GGBFS, neat FA and GGBFS blended FA binders in comparison with traditional OPC binder. The changes in length, mass and compressive strength of the specimens exposed to AMBT conditions of AS 1141.60.1 [23] were evaluated. The microstructure and reaction products of the specimens were investigated by using SEM, EDX, XRD and TGA techniques in order to understand the reaction mechanisms. Thus, the suitability of using glass

aggregate with alkali activated binders using different combinations of FA and GGBFS was evaluated.

7.2 Experimental work

7.2.1 Materials

The binder materials used in this research were OPC, FA and GGBFS. Table 7.1 shows the chemical composition of the binders; the main components of OPC and GGBFS are CaO and SiO₂, whereas FA consists predominantly of SiO₂ and Al₂O₃. The FA used in this study is classified as Class F according to ASTM C 618 [24]. The alkaline activator solutions used were 8 M NaOH solution and commercial sodium silicate solutions with chemical compositions of 29.4% SiO₂, 14.7% Na₂O and 55.9% H₂O, calculated by mass. Waste glass cullet was used as the fine aggregate. The waste glass was sieved in the laboratory before its application as the fine aggregate as per AS 1141.60.1 [23], which is given in Table 7.2. Appearances of the crushed glass particles of different sizes are shown in Fig. 7.1.

Constituents	OPC	Fly ash	GGBFS
SiO ₂	20.29	60.03	32.72
Al ₂ O ₃	5.48	22.75	13.37
CaO	63.11	3.80	41.46
Fe ₂ O ₃	2.85	6.78	0.83
K ₂ O	0.45	1.28	0.30
MgO	1.24	1.29	5.54
Na ₂ O	0.29	0.54	0.27
P_2O_5	0.17	0.89	0.01
SO ₃	2.49	0.25	4.97
TiO ₂	0.27	1.06	0.60
Mn_2O_3	0.08	0.07	0.20
SrO	0.05	0.05	0.12
Cr ₂ O ₃	0.02	0.01	0.02
ZnO	0.04		0.01

Table 7.1 Chemical compositions of the binders

Sieve	9/ hy maga		
Passing	Retained on	70 Dy mass	
4.75	2.36	10	
2.36	1.18	25	
1.18	0.60	25	
0.60	0.30	25	
0.30	0.15	15	

Table 7.2 Particle size distribution of fine aggregate as per AS 1141.60.1 [20]



Fig. 7.1 Sieved waste glass aggregate

7.2.2 Methods

7.2.2.1 Mixing, casting and curing of the specimens

In order to study the potential ASR of glass aggregate, six alkali activated mortar mixtures were prepared by using different percentages of FA and GGBFS. To compare the performance of these mixes, a control mix was prepared using OPC as the binder. The mortar mix proportions are given in Table 7.3. In the designation of the alkali activated mortar samples, F

and S stand for FA and GGBFS, respectively, followed by a number indicating its percentage. For example, the mix designation S100 represents a mixture containing 100% GGBFS in the binder. The mix containing 100% OPC as a binder was considered as the reference mix and designated as C100. A constant aggregate to binder ratio of 2.25 and activator to binder ratio of 0.47 was maintained for all the mixtures. The ratio of sodium hydroxide solution (8M) to sodium silicate solution was kept at 2. At first, all dry components were mixed for 3 minutes in a Hobart mixer and then the alkaline solution was added and mixed again for 3-4 minutes. The mixtures were poured in both $25 \times 25 \times 285$ mm prism moulds and 50 mm cube moulds. The moulds were filled in two layers of mortar and each layer was compacted using a vibrating table. They were kept at a temperature of 20 ± 3 °C and relative humidity of 65% for the next 24 hours. Then, the specimens were demoulded and steam cured at 80 °C for 22 hours. $25 \times 285 \times 285$ mm prism specimens were used for AMBT test and 50 mm cube specimens were used for compressive strength test.

Constituent mix proportions			Water to	Activator	Glass	
Mix ID	(% by weight)		cement ratio	to binder	aggregate to	
	GGBFS	FA	OPC	-	ratio	binder ratio
C100	-	-	100	0.47	-	2.25
S100	100	-	-	-	0.47	2.25
S80F20	80	20	-	-	0.47	2.25
S60F40	60	40	-	-	0.47	2.25
S40F60	40	60	-	-	0.47	2.25
S20F80	20	80	-	-	0.47	2.25
F100	-	100	-	-	0.47	2.25

Table 7.3 Mix proportions of mortar mixtures

7.2.2.2 Accelerated mortar bar test (AMBT)

AMBT of the prism specimens was conducted in accordance with AS 1141.60.1 [23]. The prism specimens were immersed in 1M NaOH solution in a sealed container at 80 °C immediately after the steam curing. After 2 hours of exposure in the NaOH solution, initial length of the mortar bars was measured using a digital length comparator. Then, the lengths of the mortar bars were recorded after 1, 5, 7, 10, 14, 21 and 28 days of exposure in the NaOH solution. The reported expansion values represent the average of results obtained from three identical specimens.

7.2.2.3 Determination of change of mass

To evaluate the change in mass of the prism specimens in AMBT condition, the mass of the mortar bars was measured using a digital weighing machine immediately after the length measurement. The specimens were wiped with a dry cloth before measuring the mass.

7.2.2.4 Determination of compressive strength

To determine compressive strength, companion mortar specimens of 50 mm cube were cast and exposed to the AMBT condition together with the mortar bar specimens. Compressive strength of the mortar specimens were tested at 14 and 28 days of exposure in AMBT condition, while the initial strength was recorded after steam curing. Compressive strength of the specimens were determined using a universal testing machine at a constant loading rate of 1.4 kN/s.

7.2.2.5 Visual inspection

The specimens were inspected regularly for appearance of surface cracks and changes in colour for 28 days of exposure in the 1M NaOH solution at 80°C.

7.2.2.6 XRD analysis

After 28 days of exposure in 1M NaOH solution at 80°C, the samples were ring milled, packed and run on the powder diffractometer instrument. XRD analysis was performed on both mortar and paste samples to investigate the reaction products using a D8 Advance Diffractometer (Bruker-AXS) instrument. The diffractometer was operated from 7° to 90° (2 θ) to scan the samples using step size of 0.015° and scanning rate of 0.5°/min.

7.2.2.7 TGA analysis

TGA was carried out using a Mettler Toledo TGA one star system analyser to study the thermal stability of the paste samples after 28 days of exposure in AMBT condition. About 30 mg of sample was placed in an aluminium crucible and heated up to 1000 °C from room temperature in an Argon environment.

7.2.2.8 Microstructural investigations by SEM and EDX

To study the microstructure of mortar specimens, SEM and EDX examinations were performed using a NEON 40EsB (ZEISS) microscopy instrument equipped with an energy dispersive X- ray analyser. Imaging was performed using a backscattered electron (BSE) with an accelerating voltage of 20 kV and aperture size of 60 μ m. The samples were cut from the mortar bars using a small diamond saw after 28 days of exposure in the NaOH solution, treated using propylene glycol and then mounted in epoxy disks of 25 mm using epoxy resin. The samples were polished using six varieties of polishing disks combined with diamond coating after 24 hours. Each step used a finer grain size than the last down to 1 μ m. The final polishing step was applied using colloidal silica solution at a pH of 11. Finally, the polished specimens were coated with carbon before the SEM and EDX analysis.

7.3 Results and discussion

7.3.1 Expansions of the mortar specimens

The expansion results of the mortar specimens are presented in Fig. 7.2. According to the Australian Standard [23], an aggregate is classified as potentially alkali silica reactive if the AMBT expansions exceed 0.1% at 10 days and 0.3% at 21 days of the test. The limiting values of AMBT expansions are shown in Fig. 7.2.


Age (days)

Fig. 7.2 Expansions of the mortar bars

It can be seen from Fig. 7.2 that the C100 mortar specimens (100% OPC) exhibited the highest expansion among all the mortars. These specimens exceeded 0.1% expansion at 5 days and 0.3% expansion at 14 days. Similarly, the alkali activated S100 mortar showed the highest expansions among the all the alkali activated mortar specimens. The S100 mortar specimens (100% GGBFS) exhibited 0.16% expansion at 10 days and 0.31% expansion at 21 days. Therefore, the expansions of C100 mortar and alkali activated S100 mortar specimens exceeded the 10-day limit of 0.1% and 21-day limit of 0.3%. Consequently, the glass aggregate can be classified as potentially reactive in both C100 and alkali activated S100 mortars bars were much less than those of the C100 mortar bars. These results are consistent with those reported by Shafaatian et al. [7] and Tennakoon [25]. Generally, OPC and GGBFS based composites

consisted of Ca rich hydration products, which are able to provide sufficient free Ca^{2+} ions in the pore solution to react with glass aggregate and form expansive compounds [13].

On the other hand, the FA and GGBFS blended alkali activated mortars as designated S80F20, S60F40, S40F60 and S20F80 showed expansions below 0.1% even after 21 days. The reason for reduced expansion is attributed to the formation of C-(N)-A-S-H (calcium-sodiumaluminosilicate hydrate) in the FA blended GGBFS alkali activated binders. This phase has a high alkali binding capacity. Also, the higher amount of aluminium in this product reduced the dissolution of silica from the reactive aggregate under alkaline environment [25-27]. Consequently, reactive aggregates showed much lower expansions in FA and GGBFS blended alkali activated binder systems as compared to the alkali activated neat GGBFS or OPC based binders. It is noticed that expansions of the alkali activated FA and GGBFS blended mortars were similar when the FA content varied from 20% to 60%. Mortar bar S60F40 showed the least expansion (0.021% after 28 days) among all the tested mortars. Although S40F60 and S20F80 mortar bars showed higher expansions than S60F40 mortar bar, the expansion values were within the 0.1% up to 28 days of testing period. Similar trend was observed by Shi et al. [28] in water glass activated GGBFS and FA blended mortars using reactive siliceous sand. The authors reported that alkalinity of the extracted solution increased with the increase of FA content (>30%) in alkali activated FA and GGBFS blended systems. Similarly, Tennakoon [25] found that both moderately reactive (gneissic origin) and reactive (hydrodacite origin) aggregates exhibited insignificant AMBT expansions (<0.1%) up to 60 days in presence of alkali activated FA and GGBFS blended binder system.

Besides, the F100 mortar bar showed 0.09% expansion at 10 days and 0.19% expansion at 21 days. Since the 10-day expansion is less than 0.1% and the 21-day expansion is between 0.1% and 0.3%, glass aggregate can be categorized as slowly reactive in alkali activated FA mortar according to AS 1141.60.1 [23]. However, this mortars showed 0.13% expansion at 14 days which is greater than the threshold expansion value (0.1%) according to ASTM C1260 [29]. It can be noted that there is no significant difference between the testing conditions of AS 1141.60.1 [23] and ASTM C1260 [29] testing methods. Consequently, this mix (F100) can be categorized as reactive as per ASTM C1260 [29] testing standard. In contrast, some studies claimed that the primary reaction product in alkali activated neat FA system is sodium aluminosilicate hydrate (N-A-S-H), which contains high alkali and aluminium but very low amount of calcium. Therefore, these studies reported that reactive aggregates used with alkali activated neat FA showed very low potential for ASR in AMBT [16-17, 19]. On the other hand,

the present study revealed that glass aggregates are less susceptible to ASR with FA and GGBFS blended alkali activated binder system than with the alkali activated binders based on FA or GGBFS alone. Therefore, the variations of the expansion results of the glass aggregate in various alkali activated binder compositions will be further justified in the later sections through other relevant experimental studies.

7.3.2 Change of mass of the mortar specimens

The variation of mass of the mortar bars with the increase of exposure duration to NaOH solution at 80 °C are plotted in Fig. 7.3. It can be seen that mass of the mortar bars increased with the increase of exposure period. Shi et al. [28, 30] noted that the mass gain is related to the amount of ASR product produced in AMBT. Since expansion occurs due to swelling of the ASR product by absorption of water, the mass gain is also considered to be related to absorption. Porosity can be expected to increase with the increase of micro cracks caused by the expansion as the exposure duration of specimens is increased. Therefore, the expansion values are plotted against mass changes in Fig. 7.4. It is seen that the S60F40 mortar bar with the lowest expansion (0.021%) exhibited the lowest increase of mass (about 1.05%) after 28 days of AMBT exposure. On the other hand, both C100 and S100 mortar bars gained greater than 1.05% mass in 10 days and showed expansions of 0.16% and 0.21%, respectively. Thus, it is noted that the expansion induced by the glass aggregates remained within the limit of 0.1% when mass gains of the C100 and S100 mortar bars were less than 1.05%. However, the FA and GGBFS blended mortars showed expansions below the 0.1% though the change of mass was about 1.2%. This result indicates that the mass gains of the FA and GGBFS blended mortars were influenced by the continued reaction of the remaining raw binder materials. On the other hand, the Ca rich mixes, i.e., C100 and S100 gained mass due to the formation of expansive ASR products under identical test condition. Therefore, it was observed that the ASR affected mortar specimens gained higher mass which is attributed to the formation of ASR product and higher absorption.



Fig. 7.3 Changes of mass of the mortar bars in AMBT



Fig. 7.4 Relationship between expansion and mass change of the mortars

7.3.3 Compressive strength

The compressive strengths of mortars after different periods of immersion in 1M NaOH solution are presented in Fig. 7.5. The results show that, C100 mortars lost about 2.5% and 4% compressive strength after 14 and 28 days of exposure in NaOH solution as compared to its initial compressive strength. On the other hand, compressive strength of all the alkali activated mortars increased considerably with the increase of exposure period except for S100 mortar. S100 mortar specimens gained very little or negligible strength at both 14 days and 28 days as compared to its initial strength. This mortar experienced some clear cracks on the surface after 14 days of AMBT exposure. Occurrence of these surface cracks are considered as the reason for negligible strength gain of the mortar S100. Shi et al [30] stated that compressive strength of the specimens decreased when expansion was high enough to cause formation of cracks.



Fig. 7.5 Compressive strengths of the mortar cubes in AMBT exposure

In this study, both C100 and S100 mortar bars exceeded 0.1% expansions before 10 days of AMBT. Their strength increase was negligible due to high surface cracks caused by high expansions. All the alkali activated mortars except S100 achieved significant strength after 14

days and 28 days of exposure. This is consistent with the observations of Shi et al. [30] and Kupwade-Patil and Allouche [17], where increases of compressive strength were reported for the mortars with relatively low expansions. The strength gain in these mortars is attributed to the continued reaction of unreacted FA particles in the AMBT condition.

7.3.4 Visual observations

Visual appearances of the mortar specimens were compared after 28 days of exposure in 1 M NaOH solution at 80 °C. It was seen that the C100 mortar bars were bright and white in colour whilst colour of the AAMs changed from dark greenish to grey white with the decrease of GGBFS content. It was also noticed that the C100 mortar bars experienced some cracks on the surface, as shown in the Fig. 7.6 (a). Similarly, S100 mortar bars exhibited significant number of cracks on the surface, as shown in the Fig. 7.6 (b). The appearance of cracks in these mortars are consistent with their high expansion values in AMBT. The FA and GGBFS blended alkali activated mortar bars, such as S80F20, S60F40, S40F60 and S20F80 did not show any crack on the surface after 28 days exposure in 1 M NaOH solution at 80 °C, as shown in Figs. 7.6 (c), 7.6 (d), 7.6 (e) and 7.6 (f), respectively. This is because these mortars showed small expansions (<0.1% expansions up to 28 days) in AMBT, as discussed in the earlier sections. However, F100 mortar specimens experienced some minor cracks on the surface after 28 days of immersion in the NaOH solution, as shown in Figs. 7.6 (g). In contrast, Kupwade-Patil and Allouche [17] did not notice cracks on the surface of alkali activated FA mortar using siliceous limestone aggregates. This may be because the siliceous limestone aggregates are less reactive than glass aggregate, as reported by Pouhet and Cyr [21]. The use of glass aggregate produced higher expansion in the neat FA system than those FA and GGBFS blended systems that resulted in some minor surface cracks.



(a) C100 mortar bar



(b) S100 mortar bar



(c) S80F20 mortar bar



(d) S60F40 mortar bar



(e) S40F60 mortar bar



(f) S20F80 mortar bar



(g) F100 mortar bar

Fig. 7.6 Visual appearances of the mortar bars after 28 days of exposure in 1 M NaOH solution at 80 °C

7.3.5 XRD analysis

The XRD analysis of the paste and mortar samples of C100, S100, S60F40 and F100 after 28 days of AMBT are shown in Fig. 7.7. The XRD results of C100 paste show that the main reaction products in C100 paste is portlandite (calcium hydroxide), which provides calcium

ions to form expansive ASR gel in the presence of reactive aggregate. It has been noticed that the intensity of portlandite peaks reduced significantly in C100 mortar as compared to the peak observed in C100 paste under the same exposure condition. This reduction of portlandite in the mortar as compared to that in the paste is attributed to the consumption of portlandite in the dissolution process of reactive silica present in glass aggregate. As a result, the C100 mortar containing glass aggregate showed the highest expansion among the all mortars. On the other hand, C-S-H and quartz phases were detected in the S100 paste and mortar samples.

CSH-Calcium silicate Hydrate (PDF 00-033-0306); M-Mullite (PDF 00-015-0776); Q-Quartz (PDF 00-046-1045); Zp-Zeolite (PDF 01-080-0699); G-Gismondine (PDF 00-020-0452); CH-Portlandie (PDF 01-078-0315); A-Analcime (PDF 01-075-8693)



Fig. 7.7 XRD of paste and mortar samples after 28 days of exposure in 1 M NaOH solution at 80°C

It has been reported that the C-S-H phase produced in the alkali activated GGBFS system does not have cross-linking 3D structure [25]. As a result, this disordered C-S-H structure can easily participate in exchange of ion under alkali environment to initiate ASR expansion. Consequently, S100 mortar bars exhibited higher expansions in AMBT. However, S100 mortar bars showed less susceptibility to the ASR as compared to the C100 mortar bars due to the absence of portlandite phase. The F100 paste sample showed the presence of zeolite, mullite and quartz (silicon dioxide) phases after 28 days of AMBT. Generally, the zeolite phase present in F100 paste indicates the formation of reaction product sodium aluminosilicate hydrate. Besides, mullite and quartz phases are detected in F100 due to the presence of unreacted FA particles since these phases are mainly present in the original FA particles [15]. From the XRD of F100 mortar sample, it can be seen that analcime phase was detected along with zeolite, mullite and quartz phases. It can also be noted that analcime phase was not detected in the XRD of F100 paste sample. This analcime phases are mainly icositetrahedral crystals that can grow rapidly to a very large size inside the matrix [17, 20]. Therefore, the formation of analcime crystals in the F100 mortar bar is considered as the likely reason for the noticeable expansion in these specimens. In S60F40 paste, sodium aluminosilicate hydrate (zeolite), calcium aluminosilicate hydrate (Gismondine) and quartz are the main detected phases. These phases (zeolite and Gismondine) are mainly hydrated aluminosilicate phases which consists of a stable cross-linking 3D structure [16, 25]. These phases can reduce the alkalinity of the pore solution due to its high alkali binding capacity [31]. By comparing the peaks observed in S60F40 paste and mortar samples, it can be seen that the detected phases are similar which proves that the available alkalis present in this binder were mostly consumed. Besides, the use of low calcium FA (40% FA in S60F40 mortar mix and 60% FA in S40F60 mortar mix) also reduced the availability of calcium into binder matrix. It is usually accepted that the presence of calcium accelerates the silica dissolution rate under alkaline environment [13]. Therefore, the highly reactive waste glass aggregate showed less expansion in alkali activated FA and GGBFS blended systems, as presented in the earlier sections.

7.3.6 TGA analysis

TGA was performed on the C100, S100, S60F40, S40F60 and F100 paste after 28 days of AMBT exposure. The results are shown in Fig. 7.8. Generally, the mass loss in hydrated cement paste occurs due to the evaporation of physically bound water from C-S-H between 50 °C and 200 °C and the mass loss between 450 °C and 550 °C is due to the dehydration of calcium hydroxide [32-33]. On the other hand, N-A-S-H is the main reaction product in alkali activated

neat FA system, C-(N)-A-S-H is the main reaction product in blended FA and GGBFS binder system and C-S-H is the main hydration product in alkali activated neat GGBFS system. It has been reported that the total mass loss at temperatures from 300 °C to 600 °C can be considered as a relative measure of the degree of reaction in the AAC [34].



Fig. 7.8 Thermogravimetric curves of paste samples after 28 days of exposure in 1 M NaOH solution at 80°C

From Fig. 7.8, it is seen that S100 paste showed the highest mass loss between 300 °C and 600 °C among all the alkali activated materials, which indicates the production of higher amount of reaction products. Consequently, this mix showed the highest compressive strength after the initial curing condition. The results also suggest that the degree of reaction decreased with the decrease of GGBFS content. As a result, S60F40 paste sample showed greater mass loss as compared to that of S40F60 paste sample. Apparently, TGA of samples does not provide any information related to the expansion results of different binder compositions except the reason behind the variations of compressive strength of different mixtures. However, one possible inference that can be made from the TGA results is the availability of greater amount of C-S-H in S100 mix which provides some calcium for the dissolution of reactive silica present in

glass aggregate. Consequently, S100 mortar bars showed higher expansions than the other alkali activated mortars. Similarly, C100 paste experienced significant amount of mass loss between 450 °C and 550 °C which indicates the dehydration of calcium hydroxide. As a result, the abundance of portlandite (calcium hydroxide) promoted the ASR severely in the binder system of C100. On the other hand, in FA and GGBFS blended systems, the available calcium is mainly consumed during the formation of C-(N)-A-S-H products. Therefore, glass aggregate showed less ASR susceptibility in FA and GGBFS blended systems as compared to the alkali activated neat FA or GGBFS binder systems.

7.3.7 Microstructural investigations by SEM and EDX

Microstructures of the C100, S100, S60F40 and F100 mortar specimens were studied after 28 days of AMBT using SEM images and corresponding EDX analysis. The microstructure of C100 reveals that, both the gaps in the aggregate-paste interface and the microcracks of aggregates were filled by a greater amount of gel, as seen in Fig. 7.9. This gel is slightly darker than the surrounding glass aggregates. EDX analysis of this gel indicated very strong peak for Si and comparatively smaller peaks for Na, Ca and Al (Fig. 7.10). The Na/Si, Al/Si and Ca/Si ratios of the gel found in the C100 mortars were 0.31, 0.02 and 0.27, respectively. The atomic ratios found in the gel of C100 mortars showed good agreement with the atomic ratios of typical ASR gel as reported in other studies [14, 33, 35]. Initially, the reactive silica present in glass aggregate-paste interface of cement mortar. Then, this gel becomes expansive by absorption of water which can introduce large stress and cracking [36-37].



Fig. 7.9 SEM image of C100 mortar after 28 days of AMBT



Fig. 7.10 EDX spectra at point A of C100 mortar

As comparing the microstructures of C100 and S100 mortars, the presence of the gel is relatively low in both the aggregate-paste interface and the microcracks of the aggregate of S100 mortar than those in the C100 mortar, as shown in Fig. 7.11. Interestingly, the elemental peaks at EDX obtained for this gel indicated very strong peak for Si and comparatively smaller

peaks for Na, Ca and Al, which is comparable with the EDX peaks obtained for the gel produced in C100, as presented in Fig. 7.12. The Na/Si, Al/Si and Ca/ Si ratios of the gel observed in the S100 mortars are 0.53, 0.05 and 0.22, respectively. It is also noted that Ca/Si ratio in ASR products of S100 mortar was slightly lower than the Ca/Si ratio of the ASR product formed in C100 mortar. In addition, it has been reported that hydration reaction products showed the highest expansions when Ca/Si ratio was between 0.20 and 0.30 [38]. This is in good agreement with the ASR expansion results presented in the earlier section (Section 7.3.1).



Fig. 7.11 SEM image of S100 mortar after 28 days of AMBT



Figure 7.12 EDX spectra at point B of S100 mortar

In S60F40 mortar, it is noticed that presence of the gel is significantly less in both the aggregate-paste interface and the micro cracks of the aggregate, as shown in Fig. 7.13. As a result, the surfaces and edges of the aggregates were almost intact in S60F40 even after 28 days exposure in the harsh AMBT environment. It can be noted that the proportion of Al is more pronounced than the Na and K peak in the gel type rim of S60F40 mortar, as shown in Fig. 7.14. The Na/Si, Al/Si and Ca/Si ratios of the gel observed in the S60F40 mortar are 0.48, 0.16 and 0.19, respectively. From the compositions of gel formed in S60F40 mortar, it is clear that this gel consisted of high Al/Si ratio and low Ca/Si ratio as compared to the gels formed in C100 and S100 mortars. Therefore, the gel formed in the S60F40 mortar is most likely C-(N)-A-S-H instead of typical ASR gel found in cement mortars. Consequently, this mortar did not show high expansion in AMBT. Besides, it was found that reaction products with low Ca/Si ratio and high Al/Si ratio showed high alkali binding capacity which leads to the reduction of available alkali ions to react with the reactive aggregate [31, 39].



Fig. 7.13 SEM image of S60F40 mortar after 28 days of AMBT



Fig. 7.14 EDX spectra at point C of S60F40 mortar

From Fig. 7.15, it is seen that a gel type rim is also formed at the aggregate paste interface and the microcracks of the aggregate of F100 mortar. The EDX of this gel confirms higher proportion of Al than Ca and Na (Fig. 7.16). This gel had Al/Si and Na/Si ratios of 0.33 and 0.46, respectively, which indicates formation of N-A-S-H as the reaction product.



Fig. 7.15 SEM image of F100 mortar after 28 days of AMBT



Fig. 7.16 EDX spectra at point D of F100 mortar

Moreover, the proportions of elements obtained for the gel formed in S60F40 and F100 mortars are different to a certain extent from those observed in typical ASR product, shown in Fig. 7.9. Consequently, the gels formed in S60F40 and F100 mortar samples are not typical ASR gel. These are mainly hydrated products (N-A-S-H and C-A-S-H), which belongs to the zeolite

phases. Similar observations are also reported by Garcia-Lodeiro et al. [20] and Tennakoon [25]. Generally, the XRD results of corresponding paste samples also provided similar information regarding the reaction products of binders S60F40 and F100. Although there is no evidence of formation of ASR type gel in F100 mortar, this mortar showed noticeable expansions in AMBT due to the formation of analcime phases as discussed in earlier section. García-Lodeiro et al. [20] stated that the analcime phases formed in alkali activated neat FA systems is able to generate internal stresses in to the matrix under AMBT condition. Therefore, this is considered as the likely reason for the noticeable expansions and minor surface cracks of F100 mortar specimens. Moreover, the gel formed in the alkali activated neat FA or FA and GGBFS blended systems is not a typical ASR type gel that is observed in alkali activated neat GGBFS or normal OPC system due to the presence of higher Al content. Therefore, glass aggregate showed very low expansions in alkali activated FA and GGBFS or normal OPC system due to alkali activated neat GGBFS or normal OPC based binder systems in AMBT as compared to alkali activated neat GGBFS or normal OPC based binder system.

7.4 Summary

According to the experimental results presented in this chapter, crushed glass fine aggregate was found reactive in OPC mortar and alkali activated GGBFS mortar. However, it was found slowly reactive in alkali activated mortars using 100% FA and non-reactive in alkali activated FA-GGBFS blended systems. The 28-day expansions of the alkali activated mortars were below 0.1% when 20% to 80% GGBFS was blended with FA. The mortars showing high expansions also showed surface cracks and reduced compressive strength by the AMBT exposure. The significant variation in ASR susceptibility of glass fine aggregate in AMBT for different binder compositions is attributed to the following reasons:

1. XRD of the samples exposed to AMBT conditions revealed that OPC or alkali activated GGBFS binders produced portlandite and C-S-H phases, which provide Ca²⁺ to generate expansive ASR gel in the presence of reactive glass aggregates. On the other hand, alkali activated FA and GGBFS binders mainly formed N-A-S-H type zeolite and C-A-S-H phases. Therefore, expansive gel was not produced in these mortars due to the lack of Ca and alkalis in the reaction product. Besides, reactive glass aggregate introduced analcime phase in alkali activated FA mortar which is considered as the likely reason for the observed expansion of the alkali activated neat FA mortar.

2. When reactive glass aggregate was used, the reaction products observed in the aggregate-paste interface of alkali activated GGBFS mortars and OPC mortars consisted of high Ca/Si ratio and low Al/Si ratio, which is comparable to the elemental compositions of typical ASR products. However, alkali activated FA-GGBFS mortars generated products of low Ca/Si and high Al/Si ratio in the presence of glass aggregate. Therefore, the presence of low Ca and high Al of these reaction products hindered the ASR potentiality of reactive glass aggregate.

7.5 References

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CHAPTER 8

EVALUATION OF THE ALKALI SILICA REACTION OF WASTE GLASS FINE AGGREGATE BY CONCRETE PRISM TESTS

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This chapter evaluates the potential ASR of waste glass cullet in alkali activated concrete, using CPT method, in comparison to that in OPC concrete. In this context, the CPT expansions, physical appearance, porosity and hydroxyl ion (OH⁻) concentration in the solution extracted from the concrete samples as well as microstructural investigation were performed.

8.1 Overview

Generally, a significant amount of alkali activator is used during the production of AAC, which may raise concerns regarding the ASR susceptibility of reactive aggregates in AAC. The most commonly used test methods to identify ASR susceptibility to aggregates in the laboratory are the AMBT, as per ASTM C1260 [1] and the CPT, as per ASTM C1293 [2]. Some published research has reported the ASR susceptibility of reactive aggregates in alkali activated materials, using either the AMBT or the CPT [3-4]. However, the findings of these studies are contradictory. For instance, Bakharev et al. [5] found that alkali activated GGBFS concrete containing reactive coarse aggregate sourced from Queensland in Australia exceeded the 0.04% expansion limit before one year in the CPT, whereas the expansion of conventional OPC concrete was below 0.04%, even one year after the test. However, Al-Otaibi [6] reported that alkali activated GGBFS concrete and OPC concrete using reactive greywacke fine aggregate both showed expansion below the 0.04% limit at one year in the CPT. Furthermore, Williamson and Juenger [7] noticed that alkali activated FA concrete containing highly reactive chert aggregate showed only a 0.006% expansion in the CPT after two years, whereas the expansion of OPC concrete reached the 0.04% expansion limit after just one month. A recent study by Rodrigue et al. [8] reported that the CPT expansion of alkali activated GGBFS concrete containing reactive Spratt aggregate was 0.151% after two years, whereas alkali activated FA concrete showed only a 0.015% expansion. Meanwhile, Garcia-Lodeiro et al. [3] and Fernandez-Jimenez et al. [9] observed that alkali activated FA mortar, with either reactive or non-reactive aggregates, exceeded the allowable expansion limit of 0.1% in the AMBT. However, Kupwade-Patil and Allouche [10] found that the expansion of alkali activated FA

mortar was much lower than that of the 0.1% expansion limit of the AMBT after 14 days. From the above literature review, it is evident that the ASR behaviour of reactive aggregates in alkali activated composites showed a wide variation in both the test methods. Therefore, more experimental research is essential in order to comprehend the ASR behaviour of reactive aggregates when used with alkali activated binders. Moreover, the CPT is usually considered more reliable than the AMBT since the samples are exposed to more realistic environmental conditions and alkali leaching is minimised due to the larger cross section of concrete prism samples [11-12]. In the AMBT, mortar bar specimens are immersed in 1 M sodium hydroxide (NaOH) solution at 80°C, which is considered to be highly aggressive and can produce unreliable results [13]. Moreover, the AMBT results of alkali activated mortar samples can be underestimated due to their autogenous shrinkage behaviour [14]. In addition, the rapid growth of zeolitic compounds in AAC under the elevated temperature conditions of the AMBT may interrupt the actual test results [9]. In contrast, the CPT involves exposure of test specimens to a much lower temperature for longer period of time, which is more realistic. In addition, CPT is commonly used to examine the ASR of both fine and coarse aggregates but in the present study the focus is on the ASR of waste glass fine aggregate.

Consequently, the potential ASR expansion of waste glass fine aggregate in alkali activated concrete has been investigated using the CPT in this chapter. Since glass contains a high amount of reactive silica and the pore solution of the alkali activated binder is highly alkaline, potential ASR is considered to be a major concern for its use as an aggregate with alkali activated binders. While a few studies have assessed the ASR susceptibility of glass aggregate when used in alkali activated materials [15], most of the existing studies have used the AMBT to evaluate the ASR of glass aggregate in alkali activated mortar [14, 16-17]. However, there is a lack of studies reporting the ASR susceptibility of glass aggregate in alkali activated concrete using the CPT in the literature. Therefore, a detailed study is essential for the assessment of the ASR of glass aggregate in alkali activated concrete using the CPT, due to the limitations of the AMBT method. Therefore, this chapter aimed to investigate the ASR susceptibility of waste glass aggregate using the CPT, where the alkali activated concrete samples were produced by using varying percentages of GGBFS and FA. The performances of the alkali activated samples were evaluated by measuring the expansions and a visual inspection of the cracks, porosity and OH⁻ ion concentration of the extracted solution, as well as a comprehensive microstructural investigation. The results from the alkali activated samples were evaluated in comparison with those from the OPC concrete specimens.

8.2 Experimental work

8.2.1 Materials

The binder materials used in this research were OPC, FA (Class F), GGBFS, waste glass cullet as fine aggregate and crushed granite as coarse aggregate. Mixture of 8 molar sodium hydroxide solution and commercial sodium silicate solution was used as alkaline activator to fabricate alkali activated samples. The particle sizes of the glass aggregate were within the designated limits of ASTM C33 [18], as presented in Fig. 8 1. The specific gravity of the fine and coarse aggregates were 2.49 and 2.65, respectively, while the fineness moduli were 3.18 and 7.60, respectively.



Fig. 8.1 Particle size distributions of glass aggregate

8.2.2 Methods

The CPT method was used to evaluate the alkali–silica reactivity in the concrete samples. The concrete mixes were designed according to ASTM C1293 [2] and the mix proportions are given in Table 8.1. In total, seven different binder combinations were used to produce concrete samples. In the designation of the alkali activated concrete samples, F and S stand for FA and GGBFS, respectively, followed by a number indicating its percentage. For example, the mix designation S100 represents a mixture containing 100% GGBFS in the binder. Similarly, the mix designation S60F40 indicates a mixture containing 60% GGBFS and 40% FA in the binder. The mix containing 100% OPC as a binder was considered as the reference mix and designated as C100. In the reference mix, the alkali content was increased to 1.25% by the inclusion of additional NaOH, as described in the testing standard. In the alkali activated mixes, the NaOH solution to sodium silicate solution ratio were 0.45 and 2, respectively. In all mixes, the slump was kept as close as possible to 80 mm to permit satisfactory compaction of the concrete in the moulds by adjusting the liquid (activator/water) to binder ratio in between 0.42 to 0.45 by mass.

	GGBFS	FA	OPC	NH*	NS**	Water	Glass	Coarse aggregate		
Mix ID							fine aggregate	20mm	10 mm	7 mm
C100	-	-	420	3.6	-	189	634	393	393	393
S100	420	-	-	63	126	-	634	393	393	393
F100	-	420	-	63	126	-	634	393	393	393
S80F20	336	84	-	63	126	-	634	393	393	393
S60F40	252	168	-	63	126	-	634	393	393	393
F60S40	168	252	-	63	126	-	634	393	393	393
F80S20	84	336	-	63	126	-	634	393	393	393

Table 8.1 Concrete mix proportions

* 8 molar sodium hydroxide solution

** Sodium silicate solution

The concrete mixes were mixed using a power-driven concrete mixer as per the guideline of ASTM C1293. At first, all the aggregates, prepared in SSD condition, were placed in the

mixing pan and mixed thoroughly for 2 minutes. Then, binder materials were placed and mixed for another 2-3 minutes. Afterwards, the premixed alkaline solution was added gradually while mixing process was in progress and the mixing process was continued for another 3 to 4 minutes. After that, three prism moulds of 75 mm \times 75 mm \times 285 mm were cast from each mixture and the moulds were filled with two layers of concrete. After pouring each layer, the moulds were compacted by rodding and by vibrating on a vibration table simultaneously. The moulds were prepared according to ASTM C1293 [2], with steel gauge studs inserted at the ends. The initial reading was taken using a digital length comparator. Following that, the samples were kept in a sealed container with water; they were elevated above the water level by 10 mm using steel racks. The container was kept in an oven at a constant temperature of 38°C throughout the test period. The subsequent length change readings were taken at seven days, 28 days and every one-month interval up to 12 months. The samples were taken out of the oven and kept in ambient conditions for 16 hours before the length measurement.

The porosity of the concrete samples was determined by conducting the volume of permeable voids (VPV) test in accordance with ASTM C642 [19]. Concrete cylinders of 100 mm diameter and 200 mm height were cast to carry out the VPV test at 28 days and after one year of CPT exposure.

The OH⁻ concentration in the solutions extracted from the concrete samples was measured according to the ex-situ leaching method, as described by Li et al. [20]. To conduct this test, concrete samples were ground at 28 days and after one year of CPT exposure to prepare 50g of powdered samples. The samples were then immediately transferred to air-tight plastic bottles and mixed with 50g of distilled water. These bottles were continuously shaken for three days at 20°C. After that, the samples were carefully filtered using Whatman filter paper to obtain 10 ml of solution from each bottle. Finally, this solution was titrated with 0.1mol/L HCl solution to measure the OH⁻ concentration. This method has successfully been used in some previous studies to study the influence of the OH⁻ concentration of the pore solution on the development of ASR in mortar samples [21-22].

Finally, microstructural analysis was conducted by SEM and EDX using a ZEISS NEON 40EsB microscopy instrument. After one year of CPT exposure, the concrete prism was cut, and representative samples were used for the SEM investigation. The sample preparation included drying, epoxy impregnating and polishing before the field emission microscopy. The images were taken at 5-10kV accelerating voltage for secondary electrons and 20kV

accelerating voltage for backscattered electrons. Furthermore, the chemical compounds of the CPT exposed concrete specimens were characterised in accordance with the XRD technique using a D8 advance (Bruker AXS) instrument.

8.3 Results and Discussion

8.3.1 Concrete prism expansions

Expansions of the concrete specimens for the increasing period of CPT are plotted in Figs. 8.2 and 8.3. Fig. 8.2 presents the expansions of the OPC based concrete and alkali activated neat FA or GGBFS concrete specimens, whereas Fig. 8.3 shows the expansions of the FA and GGBFS blended alkali activated concrete specimens. According to ASTM C1293 [2], fine aggregate with a mean concrete prism expansion equal to or greater than 0.04% after one year is classified as reactive. The 1-year expansion limits of ASTM C1293 [2] are shown by horizontal dotted lines in both figures.

It can be seen from Fig. 8.2 that the concrete specimens C100 and S100 showed one-year expansions of 0.21% and 0.13%, respectively. However, specimen F100 exhibited an expansion of only 0.02% after one year of exposure. It can also be seen that both the C100 and S100 concrete specimens exceeded the 0.04% expansion limit after 90 and 120 days, respectively. Moreover, the expansions of these specimens showed a noticeably increasing trend, even after one year. The high expansions of these specimens indicated the high ASR activity of the glass cullet with OPC and neat GGBFS alkali activated binders. However, the F100 concrete specimens showed almost stable expansions after 180 days. This observation concurred with the results reported by Li et al. [23], in which the authors found that OPC concrete and alkali activated GGBFS concrete containing reactive Spratt siliceous limestone exceeded the allowable limit of ASTM C1293 [2] before 100 days. However, the expansions of alkali activated FA concrete with reactive Spratt siliceous limestone were within the limit of ASTM C1293 [2] up to one year later.



Fig. 8.2 Expansions of OPC and alkali activated neat GGBFS or FA concrete specimens It can be seen from Fig. 8.3 that the alkali activated FA and GGBFS blended concrete specimens – S80F20, F80S20, S60F40 and F60S40 – showed expansions much lower than 0.04% throughout the test periods. In the presence of glass aggregate, the S60F40 concrete specimens showed the lowest expansions and the S80F20 concrete specimens showed the maximum expansions among all of the alkali activated FA and GGBFS blended concrete specimens. Furthermore, the expansion trends in the S60F40, F60S40 and F80S20 concrete specimens became stable after 150 days, whereas the S80F20 concrete specimens showed the lowest expansion after 300 days. In addition, it can be said that glass aggregate showed the lowest expansions with alkali activated binder systems when the combination of FA and GGBFS varied between 40-60%.



Fig. 8.3 Expansions of FA and GGBFS blended alkali activated concrete specimens

The physical appearances of the concrete specimens after one year of CPT exposure are presented in Fig. 8.4. As seen from Figures 8.4 (a) and 8.4 (b), both the C100 and S100 concrete specimens experienced visible cracks, which propagated in longitudinal and lateral directions forming a continuous network, after one year. It can be seen that the S100 concrete specimen exhibited significantly fewer cracks compared to the C100 concrete specimens. Moreover, the C100 and S100 concrete specimens showed the first crack at 180 days and 210 days, respectively. In contrast, no visible cracks were observed in the F100, F80S20, S80F20, F60S40 and S60F40 concrete specimens after one year of CPT exposure, as shown in Figs. 8.4 (c), 8.4 (d), 8.4 (e), 8.4 (f) and 8.4 (g), respectively. Therefore, glass aggregate showed innocuous expansions when used in alkali activated neat FA or alkali activated FA-GGBFS systems; however, it exhibited deleterious expansions in OPC or alkali activated neat GGBFS

binder systems. The reasons behind the variations in the expansion behaviour of glass aggregate in different binder systems are discussed in the following sections.



(a) C100 concrete specimen



(b) S100 concrete specimen



(c) F100 concrete specimen



(d) F80S20 concrete specimen



(e) S80F20 concrete specimen



(f) F60S40 concrete specimen



(g) S60F40 concrete specimen

Fig. 8.4 Visual appearances of the concrete samples after one year CPT exposure

8.3.2 Relationship between the expansions of CPT and AMBT

The ASR susceptibility of waste glass aggregate as evaluated using the AMBT is reported in Chapter 7, where the fine aggregate and binder combinations were similar to those of the present study. In the AMBT, mortar bar samples were exposed to 80°C in 1 M NaOH solution for a minimum of 14 days and the expansions were measured at various designated intervals. According to ASTM C1260 [1], the maximum allowable expansion after 14 days of AMBT exposure is 0.1%. The 14-day expansions of the mortar bars corresponding to the concrete mixtures of this study are shown in Fig. 8.5.



Fig. 8.5 14-day expansions of mortar bars in AMBT

It can be seen from Fig. 8.5 that the expansions of the C100, S100 and F100 mortar bars exceeded the allowable limit of 0.1%. In contrast, the expansions of GGBFS and FA blended alkali activated mortar bars - S80F20, F80S20, S60F40 and F60S40- were within the allowable limit of the AMBT. The expansions of the C100, S100, F100, S80F20, F80S20, S60F40 and F60S40 mortar bar samples were 0.34%, 0.21%, 0.12%, 0.05%, 0.03%, 0.019% and 0.02%, respectively. When the concrete prism expansions of Figs. 8.2 and 8.3 are compared with these mortar bar expansions, it can be seen that the expansions of waste glass fine aggregate followed the similar trends in both tests for the binder compositions of C100, S100, S80F20, F80S20, S60F40 and F60S40 mixes. Although the expansions of waste glass aggregate with the binder composition of F100 marginally exceeded the threshold value of the AMBT, they were found to be within the safe limit of the CPT. Similar expansion trends were also noticed by Li et al. [23] for Spratt limestone fine aggregate in alkali activated FA systems under both the AMBT

and the CPT. The relationship between the AMBT and CPT expansions of waste glass fine aggregate in various designated binder compositions is plotted in Fig. 8.6.



Fig. 8.6 Relationship between AMBT and CPT

Generally, the AMBT expansions are directly related with CPT expansions, as seen in Fig. 8.6. As shown in Fig. 8.6, a linear correlation could be established between the expansions of both tests that had a coefficient of determination of 0.94. Similar linear relationships between the AMBT and CPT expansions were reported by Thomas et al. [24] and Lu et al. [25]. However, the coefficients of determination were lower in the previous studies compared to those in the current study, since the data plots of the previous studies were highly scattered due to wide variation of aggregate types and binder compositions. Although the AMBT results give a quick
indication of the potential reactivity of fine aggregate, it is usually accepted that the test conditions are highly accelerated and significantly different from the real field conditions that mortar is exposed to. In contrast, the condition of the CPT is less aggressive, allowing a longer time for fine aggregate to show any reactivity. Therefore, the CPT is often recommended for confirmation of the deleterious expansion of fine aggregate shown by the AMBT.

8.3.3 Change of porosity after the CPT exposure

The VPV test results of the concrete specimens after 28 days of normal curing and after one year of CPT exposure are presented in Fig. 8.7.



Fig. 8.7 VPV results of the concrete samples

After 28 days of curing, it could be seen that OPC concrete showed relatively higher VPV than the AAC specimens. Moreover, the VPV increased when the percentages of the FA content exceeded 40% in AAC specimens. This observation is consistent with the findings of some previous studies [26-28]. It was expected that the presence of a higher amount of the FA- > 40% – would dominate the formation of N-A-S-H type binder gel, which has a lower pore

filling capacity than the C-A-S-H type binder gel, as formed in GGBFS rich alkali activated systems [29]. It can be noticed from Fig. 8.7 that the VPV of the C100 and the S100 concrete samples after one-year CPT exposure increased from 15.12% to 17.35% and 14.03% to 15.81%, respectively. This increase of porosity was attributed to the formation of a greater amount of Ca rich ASR gel from the reaction between the reactive silica of glass aggregate and the sufficient Ca ions provided by the hydrated phases of both the C100 and the S100 concrete samples [30]. This gel expanded by absorbing water from its surroundings, which led to the formation of substantial microcracks at the paste aggregate interface [24-26]. Similarly, Yurtdas et al. [31] observed that the porosity of OPC mortar samples containing reactive siliceous limestone sand increased by approximately 8% when exposed to 60°C and 95% relative humidity for 28 days, whereas the porosity of OPC mortar samples with nonreactive sand remained unchanged under similar exposure conditions and test periods. Interestingly, in this study, a decrease in porosity was observed in the S80F20, F80S20, S60F40, F60S40 and F100 concrete samples after one year of CPT exposure. In these samples, most of the free alkalis or Ca ions were consumed during the formation of N-A-S-H and C-A-S-H type reaction products. Therefore, the reactive silica of glass aggregate in these samples might have participated in the formation of an innocuous type gel instead of an expansive ASR type gel, due to the lack of free alkalis or Ca ions. This gel may have travelled freely through the interfacial transition zone and entered into the adjacent micropores of the matrix. As a result, the total porosity of these samples was significantly reduced after one year of CPT exposure. This was further substantiated by an investigation into the microstructures of these samples, as presented in Fig. 8.8. As seen in Figs. 8.8 (a) and 8.8 (b), the microstructure of both the C100 and S100 concrete samples consisted of a significant number of internal voids and cracks. However, there were significantly fewer voids and cracks in the S60F40 and F100 concrete samples, as shown in Figures 8.8 (c) and 8.8 (d). The microstructural features concurred with the VPV test results shown in Figure 8. Moreover, the specimens showing high expansions in CPT also showed an increase of porosity after CPT exposure, due to the internal stress caused by the ASR expansion that induced microcracks. In contrast, the samples showing low or allowable expansions in the CPT exhibited a decrease of porosity due to the densification of the matrix caused by innocuous reaction products.



Fig. 8.8 SEM images of the samples after VPV test

8.3.4 Change of OH⁻ ions after CPT exposure

It is generally accepted that ASR is influenced by the OH⁻ ion concentration of pore solutions, since the silica framework (Si-O-Si) of an aggregate is attacked by the OH- ion of the pore solution at the initial stage of the ASR, leading to dissolution of the silica framework [32-33]. Therefore, OH⁻ ion concentrations of the specimens were measured using the ex-situ extraction method after 28 days of normal curing and one year of CPT exposure. The results of the change

of OH⁻ ion concentrations in the extracted solution of the concrete samples are shown in Fig. 8.9.



Fig. 8.9 OH⁻ ion concentration in the extracted solution of the concrete samples

Fig. 8.9 shows that the OH⁻ ion concentration in the C100 sample was less than that of the alkali activated samples after 28 days of normal curing. This was because of some NaOH solution may have remained unused when the alkali activated samples were cured under normal conditions for 28 days. Similarly, Shi et al. [21] noticed that the OH⁻ ion concentration in the pore solution of OPC mortars was much lower than that of alkali activated GGBFS mortars after water or steam exposures at various stages. Moreover, it could be seen that OH⁻ ion concentration was the highest in the S100 sample after 28 days of initial curing. This was consistent with the findings reported by Shi et al. [22]. After one year of CPT exposure, all samples exhibited a decrease in OH⁻ ion concentration, which was more prominent in the alkali

activated concrete samples compared to the OPC concrete samples. For example, the C100 sample showed a 1.89% decrease of OH⁻ ion concentration, whereas alkali activated concrete samples exhibited between 5% to 11% decrease after one year of CPT exposure. This was primarily attributed to the absence of the Portlandite phase in the alkali activated binder systems, since this phase acted as a buffer to maintain the pH of the pore solution by supplying OH⁻ ions. Therefore, the C100 concrete sample showed significantly higher expansion compared to the AAC samples, as shown in Figs. 8.2 and 8.3. Furthermore, the decrease of OH⁻ ion concentrations in the F100, S80F20, F80S20, S60F40 and F60S40 concrete samples were 9.94%, 9.77%, 10.06%, 11.45% and 10.30%, respectively whereas, the decrease of OH⁻ ion concentrations in the S100 concrete sample was 6.15%. The reason for this is that the reaction product formed in the F100, S80F20, F80S20, S60F40 and F60S40 samples was mainly C-(N)-A-S-H or N-A-S-H type gels, which binds a higher number of Na ions in their crosslinking networks [34]. However, the main reaction product of the S100 concrete samples is C-S-H, which is relatively less stable compared to the C-(N)-A-S-H or N-A-S-H reaction products that led to an increase of the availability of Ca and OH⁻ ions in the pore solution. This finding supported the higher expansions obtained for the S100 sample compared to the expansions of other alkali activated samples - F100, S80F20, F80S20, S60F40 and F60S40 after CPT exposure. Although the OH⁻ ion concentration of the S100 concrete sample was significantly higher than the C100 concrete sample at both testing ages, it exhibited lower expansion than the C100 concrete sample. This may have been due to the availability of a greater amount of Ca ions in the pore solution of the C100 concrete compared to the S100 concrete. Gruskovnjak et al. [35] found that the Ca ion concentration in alkali activated GGBFS systems was about ten times lower than that of the OPC based systems.

8.3.5 Microstructure investigation

From the above discussion, it is evident that the use of waste glass as fine aggregate exhibited significant expansion of concrete when the binder consisted of 100% OPC in conventional concrete – C100 – or 100% GGBFS in AAC – S100. However, the expansions were significantly less in alkali activated concrete samples formulated with 100% FA – F100 – or blends of GGBFS and FA – F80S20, S80F20, F60S40 and S60F40 – as the binder. The expansions at the end of the one-year testing period of concrete prisms using these binder compositions were considered not deleterious since the expansions were within the 0.04% limit of the ASTM C1293 [2]. Considering these findings, the C100, S100, F100 and S60F40 concrete samples were selected for the microstructure investigations to further understand the

significant variations in the expansions of waste glass in these binder compositions. Microstructures of the C100, S100, F100 and S60F40 concrete samples were investigated using SEM, EDX and XRD analyses. The SEM images and representative EDX spectra of the C100, S100, F100 and S60F40 specimens are shown in Figures 8.10 to 8.22. Fig. 8.10 shows that the glass aggregates were severely damaged due to the formation of a substantial amount of ASR induced cracks, which were more prominent on the surface of the glass aggregates compared to the surrounding paste matrix or aggregate paste interface. Guo et al. [36] and Khan et al. [16] observed a similar type of microstructure in OPC mortar samples containing glass aggregate. The element mapping showed high concentrations of Si and Ca in the microstructure of C100, as shown in Fig. 8.11, where the red and yellow colours represent the concentration of Si and Ca, respectively. The presence of the Si element was distributed to the area of glass aggregate, whereas Ca was mainly distributed at the paste matrix area. Moreover, EDX analysis confirmed that the chemical compositions of the products formed on the ASR damaged aggregate (C1) and aggregate paste interface (C2) of the C100 sample consisted of higher percentages of both Ca and Na, as presented in Table 8.2. It can be seen that the Ca/Si and Na/Si ratios were 0.28 and 0.37 at C1 location, and 0.34 and 0.46 at C2 location. In addition, the morphology of the gel formed in C1 and C2 was mainly either rod or sword type, as shown in Fig. 8.12. The atomic ratios and the morphologies of the gel formed in C1 and C2 locations of the C100 sample showed significant similarities with the atomic ratios and morphologies of typical ASR products described in some previous studies [16, 37].



Fig. 8.10 SEM image of C100 concrete sample

Table 8.2 Chemical co	ompositions analysis	s results of the concrete	samples (atomic %)
	· · · · · · · · · · · · · · · · · ·		~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~

Mix ID	Analysed points ID	0	Si	Ca	Na	Al	Ca/Si	Na/Si	Al/Si
C100	C1	66.8	19.4	5.5	7.1	0.5	0.28	0.37	0.03
	C2	65.6	18.1	6.2	8.3	0.9	0.34	0.46	0.05
S100	S 1	64.6	18.9	4.7	10.4	1.4	0.25	0.55	0.07
	S2	64.7	17.9	5.1	10.8	1.5	0.28	0.60	0.08
F100	F1	64.0	20.1	2.3	9.1	4.5	0.11	0.45	0.22
	F2	61.3	23.2	1.3	8.1	5.5	0.06	0.35	0.24
S60F40	F3	58.2	24.1	1.8	10.5	4.9	0.07	0.44	0.20
	S 3	63.9	19.3	3.7	9.7	3.3	0.19	0.50	0.17
	S4	60.9	20.9	3.8	10.7	3.7	0.18	0.51	0.18



Fig. 8.11 EDX mapping in the microstructure of C100 concrete sample



Fig. 8.12 Morphology of ASR gel in C100 concrete sample

Figure 8.13 shows that microstructural changes in the S100 sample due to the ASR of glass aggregates were very similar to those observed in the microstructure of the C100 sample. Moreover, Fig. 8.14 and Table 8.2 show that the atomic ratios and morphology of the ASR products observed in the S100 sample were very close to those found in the C100 sample. This was mainly attributed to a high concentration of Ca in the binder system of the S100 sample as confirmed by the EDS mapping in the microstructure, as shown in Fig. 8.15. Furthermore, the XRD results confirmed the existence of Ca dominant hydrated phases, such as Portlandite (Ca[OH]₂), Calcium carbonate (CaCO₃), Gismondine (CaAl₂Si₂O₈·4H₂O) and Calcium silicate hydrate (Ca₈Si₃O₁₄·3H₂O) in the CPT exposed C100 and S100 concrete samples, as presented in Fig. 8.16. This was because both the OPC and GGBFS samples contained significant amounts of CaO. The Ca dominant hydrated phases of the C100 and the S100 samples promoted the dissolution of silica to form expansive Ca rich ASR gel. As a result, the expansions of the C100 and the S100 concrete samples exceeded the safe limit before one year of CPT exposure, as shown in Fig. 8.2.



Fig. 8.13 SEM image of S100 concrete sample



Fig. 8.14 Morphology of ASR gel in S100 concrete sample



Fig. 8.15 EDX mapping in the microstructure of S100 concrete sample



Fig. 8.16 XRD results of C100 and S100 concrete samples

Meanwhile, any significant deterioration or damage due to ASR of glass aggregate was not observed in the interior of the glass aggregate or the aggregate paste interface area of the F100 sample, as shown in Fig. 8.17. However, some microcracks could be seen at the surface of the glass aggregate of the F100 sample. The presence of these microcracks in the glass aggregate can be referred to as the crushing of waste glass [38], since the expansions of the F100 sample were insignificant until one year of CPT exposure. Moreover, the EDX mapping showed that the paste matrix of the F100 sample contained a higher intensity of the Al element and a lower intensity of the Ca element, as presented in Fig. 8.18. Consequently, it can be said that the alkali silica gel formed in the aggregate paste interface of F100 sample was not a typical expansive Ca rich ASR gel since the Ca/Si and Al/Si ratios of this gel (points F1, F2 and F3) ranged from 0.06 to 0.11 and 0.20 to 0.24, respectively, as can be seen in Table 8.2. This gel was a mainly N-A-S-H or N-(C)-A-S-H type innocuous gel that can travel around the aggregate paste interface due to its low viscosity [39], as seen in Fig. 8.19. This hypothesis agreed with the XRD results shown in Fig. 8.20, which indicated that the major hydrated phases of the CPT

exposed F100 sample were Zeolite P1 ($Na_6Al_6Si_{10}O_{32}$ ·12H₂O), Albite ($NaAlSi_3O_8$) and Anorthite ($CaAl_2Si_2O_8$). Moreover, the presence of a significant amount of Al content in the hydrated phase of the F100 sample may have restricted the dissolution of the aggregates by providing a protective layer of aluminosilicate [40-41]. Therefore, the glass aggregates were almost intact in F100 concrete even up to one year of CPT exposure, as shown in Figure 8.17. Consequently, one-year CPT expansions of the F100 concrete sample were significantly lower compared to the S100 and C100 concrete samples, as shown in Fig. 8.2.



Fig. 8.17 SEM image of F100 concrete sample



Fig. 8.18 EDX mapping in the microstructure of F100 concrete sample



Fig. 8.19 Aggregate paste interface of F100 concrete sample



Fig. 8.20 XRD results of F100 and S60F40 concrete samples

Fig. 8.21 reveals that there was no evidence of ASR induced deterioration or damage in glass aggregate or the aggregate paste interface of the S60F40 concrete sample, which was similar to the F100 concrete sample. Although the intensity of the Ca element in the paste matrix of the S60F40 sample was higher than that observed in F100 sample, as seen in Fig. 8.18 and Fig. 8.22, the S60F40 concrete sample showed lower expansion than the F100 concrete sample. However, this Ca element was mostly engaged in a strong and stable network of calcium aluminosilicates, such as Gismondine (CaAl₂Si₂O₈:4H₂O) and Anorthite (CaAl₂Si₂O₈), in the presence of sufficient Al, which was confirmed by the XRD analysis, as shown in Fig. 8.20. Such phenomena have also been reported by Rodrigue et al. [8] for the microstructure of alkali activated FA and GGBFS concrete associated with reactive Spratt aggregate. Therefore, it can be said that the porosity and OH⁻ ion concentration results as discussed in the earlier sections were mainly responsible for the slight difference in expansions between the F100 and S60F40 concrete samples. Furthermore, the Ca/Si and Al/Si ratios of the gel formed at the aggregate paste interface (points S3 and S4) of the S60F40 sample were 0.19, 0.18, 0.17 and 0.18, respectively (Table 8.2), which were significantly different from those observed in the S100 or C100 concrete samples. Overall, the microstructural observations of the CPT exposed samples showed a positive correlation with the microstructural observations of the AMBT exposed samples, as reported in the Chapter 7.



Fig. 8.21 SEM image of S60F40 concrete sample



Fig. 8.22 EDX mapping in the microstructure of S60F40 concrete sample

8.4. Summary

According to the experimental results presented in this chapter, the one-year CPT expansions of glass fine aggregate in OPC concrete and alkali activated GGBFS concrete specimens were 0.21% and 0.13%, respectively; whereas, the expansions of the alkali activated FA and FA-

GGBFS concrete specimens were below 0.04%. The alkali activated FA-GGBFS concrete made using 60% GGBFS and 40% FA showed an expansion of only 0.015% after one year. The CPT expansions of the current study showed a positive correlation with the previously determined AMBT expansions, using the same glass aggregate. The significant variation in ASR susceptibility of glass fine aggregate in CPT for different binder compositions is attributed to the following reasons:

- After CPT exposure, OPC concrete and alkali activated GGBFS concrete specimens with waste glass aggregate experienced pattern or mapping type cracks on the surface. However, no such cracks occurred in the alkali activated FA or the FA-GGBFS concrete specimens.
- 2. The porosity of the OPC concrete and alkali activated GGBFS concrete specimens increased after CPT exposure due to the formation of a higher amount of expansive ASR products, which introduced micro cracks. However, a significant decrease in porosity was observed in the alkali activated FA and FA-GGBFS concrete specimens. This was attributed to the formation of higher amounts of non-expansive reaction products that could be accommodated in the voids and cracks.
- 3. A significant decrease in the OH⁻ concentration was found in the solution extracted from the alkali activated FA concrete and alkali activated FA-GGBFS concrete samples when exposed to the CPT conditions. This helped to restrict the dissolution of the silica framework of glass aggregate in these samples.
- 4. The SEM images clearly demonstrated that the surface of glass aggregate and the paste-aggregate interface in OPC concrete and alkali activated GGBFS concrete were severely damaged due to the ASR. The EDS and XRD results indicated that the high concentration of Ca element in the binder phase of both the OPC concrete and alkali activated GGBFS concrete mainly promoted the ASR of glass aggregate in these specimens. However, the glass aggregates were found to be intact or undamaged in the SEM images of the alkali activated FA and FA-GGBFS concretes. The presence of a significant amount of the Al element in the binder matrix of these samples may have restrained the ASR of glass aggregate. Furthermore, the composition of the gels formed in the aggregate surface and aggregate-paste interface of these samples were different to those of ASR affected samples.

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CHAPTER 9

CONCLUSIONS AND RECOMMENDATIONS

The primary focus of this thesis was to uncover the feasibility of waste glass as a partial precursor material and fine aggregate for producing AAC. The entire experimental work was conducted in two important phases. In the first phase of the experimental investigation, GP was used as 10%, 20% 30% and 40% replacement of FA (Class F) in alkali activated FA and FA-GGBFS mortars. The effects of waste glass powder as a partial binder on workability, mechanical properties and durability of alkali activated FA and FA-GGBFS mortars were investigated. Later, the efficacy of waste glass cullet as fine aggregate was investigated in the second phase of the experimental program. In this regard, waste glass culet was used as 25%, 50%, 75% and 100% replacement of natural sand in alkali activated FA-GGBFS mortars. The influence of waste glass cullet as fine aggregate on workability, mechanical properties and durability of alkali activated FA-GGBFS mortars. The influence of waste glass cullet as fine aggregate on workability, mechanical properties and durability of alkali activated FA-GGBFS mortars. The influence of waste glass cullet as fine aggregate on workability, mechanical properties and durability of alkali activated FA-GGBFS mortars. The influence of waste glass cullet as fine aggregate on workability, mechanical properties and for a program and some significant recommendations for the future studies are presented in this chapter.

9.1 Conclusions

The following conclusions are drawn from the experimental program of this thesis:

(a) Effects of using GP as a partial precursor material:

- 1. The inclusion of GP as a partial replacement of FA reduced the workability of alkali activated FA and FA-GGBFS mortars. The reduction of workability for using GP is attributed to its high specific surface and angular morphology.
- 2. The compressive strength of the alkali activated FA mortars gradually decreased with the rise of FA replacement rate by GP. However, the compressive strength of the FA-GGBFS mortars significantly increased for using 10%-20% GP and then the strength slightly declined with the further increase of GP beyond 20%. When GP was added as a replacement of FA in FA-GGBFS binder system, the additional Si, Na and Ca elements supplied by the GP enhanced the polymerization process and thus increased compressive strength. However, the increase of GP content beyond 20% affected the polymerization process adversely due to the shortage of required Al content.

Consequently, compressive strength decreased for using GP content higher than 20% in the FA-GGBFS binder system.

- 3. The alkali activated mortars containing various percentages of GP exhibited higher drying shrinkage than that of mortars without GP. This phenomenon is attributed to the slow dissolution and impermeable characteristics of GP particles which introduced capillary network with sufficient evaporable water into the matrix. Besides, the use of GP as a partial replacement of FA increased the Na/Si and Ca/Si ratios that led to the enhancement of moisture movement in the aluminosilicate products.
- 4. Partial replacement of FA with GP did not bring any significant improvement to the sorptivity, porosity and chloride permeability properties of FA mortars. However, the inclusion of 10%-20% GP as a partial binder in FA-GGBFS mortars was found beneficial to reduce the sorptivity, porosity and chloride permeability. The sorptivity coefficients of FA-GGBFS mortars with 10% and 20% GP were 3.09 × 10⁻² mm/s^{1/2} and 3.06 × 10-2 mm/s^{1/2}, whereas the sorptivity coefficient of FA-GGBFS mortar without GP was 3.91 × 10-2 mm/s^{1/2}. Similarly, the porosity of FA-GGBFS mortars containing 10% and 20% GP were 14.8% and 14.6%, whereas the porosity of FA-GGBFS mortar without GP was 15.6%. Furthermore, it was found that the chloride permeability of FA-GGFS mortars reduced by 6.07% and 13%, respectively, when 10% and 20% FA was replaced with GP.
- 5. Microstructural examinations by SEM, EDX, XRD, and TGA analysis demonstrated that the use of GP as a partial binder in FA-GGBFS blended system enhanced the formation of primary reaction products such as C-S-H, C-A-S-H and N-A-S-H gels that led to a dense and compact microstructure. However, when a higher dosage of GP (>20%) was used, a significant amount of GP remained undissolved that affected the initial reaction kinetics adversely as well as induced voids and microcracks in the matrix.
- 6. The use of 10% to 20% GP as a replacement of FA significantly improved the acid resistance performance of alkali activated FA-GGBFS mortars. After 1 year of exposure in H₂SO₄ acid, the mortar containing 20% GP showed 13.2% mass loss and 22.1% strength loss, whereas the mass and strength losses of the reference mortar were 14.1% and 31.2%, respectively. In the case of 1 year exposure in HCl acid, 9.1% mass loss and 10.6% strength loss were observed for the mortars with 20% GP, while the control mortar exhibited 13.3% mass loss and 27.6% strength loss. The mass and strength losses increased with the increase of GP content above 20% for both types of

acid exposure. Microstructural examinations using XRD, SEM and EDX analysis confirmed that the use of 10% to 20% GP as a partial precursor densified the microstructure by enhancement of the polymerization mechanism, which reduced the ingress of acid solutions into the matrix. Consequently, the samples with 10% to 20% GP showed minimal trace of depolymerized phases while exposed in acid solutions as compared to the control sample.

(b) Influence of waste glass cullet as fine aggregate:

- Natural sand replacement by waste glass fine aggregate showed increase of the workability of freshly mixed alkali activated FA-GGBFS mortar with the increase of waste glass fine aggregate content up to 100%. This is attributed to the smooth surface texture, less liquid demand and high fineness modulus of waste glass cullet.
- 2. Compressive strength of the alkali activated FA-GGBFS mortars gradually decreased with the increase of waste glass fine aggregate. This is attributed to the smooth surface texture of glass cullet that tends to weaken the bond at the ITZ and hence a reduction of compressive strength. Consequently, the use of glass fine aggregate as 25% replacement of natural sand can considered feasible in terms of compressive strength.
- 3. The drying shrinkage values of the alkali activated FA-GGBFS mortar samples containing 25% to 100% waste glass fine aggregate were less than that of control sample. This is attributed to low water absorption and high elastic modulus characteristics of glass fine aggregate.
- 4. The inclusion of 25% to 100% waste glass fine aggregate showed significant improvement on the sorptivity and chloride permeability properties of the alkali activated FA-GGBFS mortars. The positive influence of using glass fine aggregate on these properties is attributed to the impermeable nature of glass aggregate. However, the porosity of mortars was found to increase with the rise of waste glass fine aggregate. This phenomenon is attributed to the irregular shape of glass particles that tends to increase the internal voids.
- 5. The alkali activated FA-GGBFS mortars containing 25% to 100% waste glass fine aggregate showed higher residual compressive strength than that of mortars with natural sand after exposure to 600°C and 800°C temperatures. SEM images revealed that glass fine aggregate softened partially when exposed at 800°C that provided a good connection with the paste matrix at the ITZ. Consequently, the use of glass fine aggregate enhanced high temperature resistance of alkali activated samples.

- 6. The alkali activated FA-GGBFS mortars with various amounts of waste glass fine aggregate experienced relatively higher physical damages and losses of mass and compressive strength when exposed to H₂SO₄ and HCl acid solutions for 1 year as compared to the mortar specimens using natural sand. This is attributed to the smooth surface texture and angular shape characteristics of the glass aggregate which led to loose bonding at the ITZ as well as increased porosity, as revealed by SEM images. Overall, the percentages of the mass and compressive strength losses of the mortars containing 25% to 50% glass aggregate were comparable with those of the control mortar.
- 7. According to the expansion measurements by AMBT, the waste glass fine aggregate was found highly reactive when used in OPC mortar and alkali activated GGBFS mortar. However, it was found slowly reactive when used in alkali activated FA mortar. Also, waste glass fine aggregate exhibited non-reactive behaviour when used in FA-GGBFS blended alkali activated mortars. The lowest expansion was found for the mortar specimen associated with 60% FA and 40% GGBFS in the binder and waste glass fine aggregate content of 100%. These observations are well aligned with the physical observations, mass change and compressive strength of AMBT exposed mortar samples. Besides, the microstructural observations revealed that the presence of Ca based hydrated phases in OPC or alkali activated GGBFS promoted the production of expansive ASR products around the glass aggregate, which led to extensive expansion resulting in cracks. The lower expansions of waste glass fine aggregate in FA and FA-GGBFS binder systems is attributed to the formation of hydrated phases with low Ca and free alkali elements as well as the presence of high Al element. Furthermore, elemental analysis by EDS indicated that the characteristics of the products formed at the ITZ of FA and FA-GGBFS binders were different from that of products formed in the ASR affected samples.
- 8. The ASR behaviour of waste glass fine aggregate as evaluated by CPT showed a positive correlation with results obtained by AMBT. According to the CPT results, OPC and alkali activated GGBFS concrete specimens containing waste glass fine aggregate exceeded the 0.04% expansion limit after 1-year test period. On the other hand, the expansions of alkali activated FA and FA-GGBFS concretes with waste glass fine aggregate were well below this threshold value after 1-year test period. The lowest expansion was found for the concrete specimen associated with 60% FA and 40% GGBFS in the binder and waste glass fine aggregate content of 100%. These findings

showed consistency with the physical observation, porosity and OH⁻ concentration in pore solution of the test samples, when exposed to the CPT environment for 1-year. The EDS mapping and XRD results confirmed that the high concentration of Ca element in the binder phase of OPC and alkali activated concrete promoted the ASR of waste glass fine aggregate. However, relatively high concentration of Al element and low concentration of Ca element were traced in the binder phase of FA and FA-GGBFS samples that may have positive influence to restrict the ASR of the glass fine aggregate.

9.2 Recommendations for future study

This thesis has dealt with an experimental investigation on feasibility of using waste glass as a partial precursor material and fine aggregate for making AAC. The influence of waste glass powder (as a partial precursor material) and glass cullet fine aggregate on the fresh and mechanical properties, durability and microstructural aspects of AAC were evaluated from this research. The findings of this thesis manifest that waste glass has a considerable potential for use as a partial precursor and fine aggregate in preparation of AAC due to its acceptable physical, chemical and microstructural characteristics. However, there are few related aspects that were not covered in this thesis due to the limitations of time frame and equipment that can be considered in future as follows:

- This research investigated the efficacy of GP as a binder component in alkali activated FA and FA-GGBFS mortars cured at ambient conditions, where FA was replaced partially using GP. Similar research can be done for alkali activated MK and MK-GGBFS mortars, where MK would be substituted partially by GP.
- In this research, NaOH and Na₂SiO₃ solutions were used as alkaline activators to activate the raw materials. Further studies can be performed using other activators such as KOH and K₂SiO₃.
- 3. This study mainly focused on the mechanical and durability performance of AAC containing GP and waste glass fine aggregate. Dynamic properties such as ultimate stress, strain, toughness etc. can be evaluated in future for the mixtures of this thesis.
- In this study waste glass cullet has been considered as fine aggregate for making AAC. Further research can be done to evaluate the suitability of waste glass cullet as coarse aggregate in preparation of AAC.
- This study investigated the performance of GP and waste glass fine aggregate in AAC separately. A study can be done considering the concurrent use of GP and waste glass fine aggregate.

 This study has been limited to mechanical and durability properties. Further studies can be done on life cycle assessment and modelling aspect of the use of recycled glass in alkali activated composites.

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APPENDIX A: Images of experiments



Fig. A.1 Collection of materials



Fig. A.2 Preparation of mortar



Fig. A.3 Mortar flow measurement



Fig. A.4 Compressive strength test



Fig. A.5 Curing of samples for drying shrinkage test



Fig. A.6 Pore solution alkalinity testing



Fig. A.7 Preparation of samples for sorptivity test


Fig. A.8 Storage of samples for acid resistance test

APPENDIX B: Experimental Data

Miy ID	Compressive strength (MPa)							
	7 days	14 days	28 days	90 days				
G0F	18.7	28.25	36.65	40.55				
G10F	17.2	26.85	35.75	39.95				
G20F	15.3	25.35	34.65	38.45				
G30F	13.6	23.15	32.65	37.25				
G40F	12.4	21.25	30.85	35.05				

Table B.1 Compressive strength of FA mortars containing 10% to 40% GP

Table B.2 Compressive strength of FA-GGBFS mortars containing 10% to 40%GP

Mix ID	Compressive strength (MPa)							
	7 days	14 days	28 days	90 days				
G0FS	46.8	63.3	71.1	76.8				
G10FS	48.3	67.9	75.5	80.9				
G20FS	49.4	70.5	78	85.3				
G30FS	43.2	59.7	67.8	74.1				
G40FS	39.7	53.9	62.5	68.3				

Table B.	3 Flow	values o	f the a	ılkali	activated	FA	and FA-	-GGBFS	mortars	with GP
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Mix ID	Flow (%)
G0F	147.1
G10F	140.0
G20F	134.3
G30F	127.3
G40F	121.6
G0FS	116.2
G10FS	110.4
G20FS	105.6
G30FS	100.7
G40FS	94.8

Table B.4 Sorptivity coefficient of the alkali activated FA and FA-GGBFS mortars with GP

Mix ID	Sorptivity coefficient (×10 ⁻² mm/S ^{1/2})
G0F	4.66
G10F	5.44
G20F	5.81
G30F	5.97
G40F	6.20
G0FS	3.91
G10FS	3.09
G20FS	3.06
G30FS	3.68
G40FS	4.38

Table B.5 Porosity of the alkali activated FA and FA-GGBFS mortars with GP

Mix ID	Porosity (%)
G0F	17.1
G10F	17.3
G20F	17.4
G30F	17.6
G40F	17.9
G0FS	15.7
G10FS	14.8
G20FS	14.6
G30FS	15.4
G40FS	16.5

Table B.6 RCPT results of the alkali activated FA and FA-GGBFS mortars with GP

Mix ID	Test duration (hours)	Charge passed (Coulombs)
G0F	3.7	-
G10F	3.2	-
G20F	2.9	-
G30F	2.8	-
G40F	2.2	-
G0FS	6	4288
G10FS	6	3915
G20FS	6	3727
G30FS	6	4597
G40FS	6	4935

Mix ID	Compressive strength (MPa)							
	7 days	14 days	28 days	90 days				
AAMG0	46.8	63.3	71.1	76.8				
AAMG25	44.5	60.5	69.5	75.3				
AAMG50	42.5	58.1	68.3	74.1				
AAMG75	42.0	56.9	67.6	73.6				
AAMG100	41.2	55.8	65.7	73.1				

Table B.7 Compressive strength of mortars containing 25% to 100% wasteglass fine aggregate

Table B.8 Expansions (%) of the mortar bars containing waste glass fine aggregate in AMBT

Mix ID	1 day	5 days	7 days	10 days	14 days	21 days	28 days
C100	0.01	0.09	0.13	0.21	0.31	0.48	0.6076
S100	0.01	0.08	0.11	0.16	0.21	0.31	0.38
F100	0.0088	0.035	0.0534	0.08	0.12	0.18	0.22
S80F20	0.01	0.02	0.029	0.039	0.05	0.065	0.08
S60F40	0.0074	0.0128	0.0174	0.0188	0.0192	0.0198	0.021
S40F60	0.0084	0.015	0.0185	0.0199	0.024	0.0258	0.031
S20F80	0.0016	0.018	0.0195	0.025	0.029	0.0382	0.045

Mix ID	7	30	60	90	120	150	180
	day	days	days	days	days	days	days
C100	0.0095	0.0220	0.0320	0.0590	0.0800	0.1200	0.1320
S100	0.0073	0.0152	0.0190	0.0310	0.0497	0.0552	0.0804
F100	0.0055	0.0100	0.0120	0.0170	0.0180	0.0210	0.0224
S80F20	0.0061	0.0130	0.0150	0.0210	0.0225	0.0235	0.0250
S60F40	0.0030	0.0070	0.0074	0.0100	0.0110	0.0135	0.0138
S40F60	0.0046	0.0081	0.0085	0.0112	0.0130	0.0164	0.0169
S20F80	0.0051	0.0090	0.0095	0.0130	0.0170	0.0180	0.0190

Table B.9 Expansions (%) of the concrete samples containing waste glass fineaggregate in CPT (7 days to 180 days)

Table B.10 Expansions (%) of the concrete samples containing waste glass fineaggregate in CPT (210 days to 360 days)

Mix ID	210	240	270	300	330	360
	days	days	days	days	days	days
C100	0.1410	0.1662	0.1790	0.1990	0.2020	0.2110
S100	0.0850	0.0930	0.0960	0.1100	0.1250	0.1290
F100	0.0225	0.0236	0.0235	0.0238	0.0241	0.0244
S80F20	0.0262	0.0269	0.0270	0.0273	0.0275	0.0277
S60F40	0.0141	0.0140	0.0142	0.0143	0.0146	0.0149
S40F60	0.0171	0.0175	0.0183	0.0181	0.0183	0.0186
S20F80	0.0195	0.0201	0.0203	0.0206	0.0209	0.0211

APPENDIX C: Attribution of research outputs

Article 1

1. M. N. N. Khan, J. C. Kuri and P. K. Sarker. Effect of waste glass powder as a partial precursor in ambient cured alkali activated fly ash and fly ash-GGBFS mortars. Journal of Building Engineering, 34 (2020) 101934.

Authors and full affiliations:

M.N.N. Khan, PhD student, School of Civil and Mechanical Engineering, Curtin University, WA.

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Co-author attribution: J.C. Kuri and P.K. Sarker

Name of Co- author	Literature review	Experimental design/idea	Data collection	Data analysis	Discussion	Paper writing		
J.C. Kuri								
I acknowledge that these represent my contribution to the above research output.								
(Signature)								
P.K. Sarker		\checkmark						
I acknowledge that	these represen	t my contribution	on to the above	e research out	put.			
(Signature)								

Article 2 and 3

2. M. N. N. Khan and P. K. Sarker. Effect of waste glass fine aggregate on the strength, durability and high temperature resistance of alkali-activated fly ash and GGBFS blended mortar. Construction and Building Materials, 263(2020) 120177.

3. M. N. N. Khan and P. K. Sarker. Alkali silica reaction of waste glass aggregate in alkali activated fly ash and GGBFS mortars. Materials and Structures, 52, 93 (2019).

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Co-author attribution: P.K. Sarker

Name of Co-	Literature	Experimental	Data	Data	Discussion	Paper writing
author	review	design/idea	collection	analysis		
P.K. Sarker						
I acknowledge that	these represen	t my contributio	on to the abov	e research out	put.	
(Signature)						

Article 4

M. N. N. Khan, A. K. Saha and P. K. Sarker. Evaluation of the ASR of waste glass fine aggregate in alkali activated concrete by concrete prism tests. Construction and Building Materials, 266 (2021) 121121.

Authors and full affiliations:

M.N.N. Khan, PhD student, School of Civil and Mechanical Engineering, Curtin University, WA.

A.K. Saha, Adjunct Research Associate, School of Civil and Mechanical Engineering, Curtin University, WA.

P.K. Sarker, Associate Professor, School of Civil and Mechanical Engineering, Curtin University, WA

Name of Co- author	Literature review	Experimental design/idea	Data collection	Data analysis	Discussion	Paper writing		
A.K. Saha					\checkmark			
I acknowledge that these represent my contribution to the above research output.								
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P.K. Sarker		N		λ	V	N		
P.K. Sarker $$ $$ $$ I acknowledge that these represent my contribution to the above research output.								

Co-author attribution: A.K. Saha and P.K. Sarker

<u>Article 5</u>

M. N. N. Khan, A. K. Saha and P. K. Sarker. Reuse of waste glass as a supplementary binder and aggregate for sustainable cement-based construction materials: A review. Journal of Building Engineering, 28 (2020) 101052.

Authors and full affiliations:

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P.K. Sarker, Associate Professor, School of Civil and Mechanical Engineering, Curtin University, WA

Name of Co- author	Literature review	Experimental design/idea	Data collection	Data analysis	Discussion	Paper writing		
A.K. Saha					\checkmark			
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P.K. Sarker						\checkmark		
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(Signature)								

Co-author attribution: A.K. Saha and P.K. Sarker

<u>Article 6</u>

M. N. Khan, J. C. Kuri and P. K. Sarker. Sustainable use of waste glass in alkali activated materials against H_2SO_4 and HCl acid attacks. Cleaner Engineering and Technology, 2020 (Under Review).

Authors and full affiliations:

M.N.N. Khan, PhD student, School of Civil and Mechanical Engineering, Curtin University, WA.

J.C. Kuri, PhD student, School of Civil and Mechanical Engineering, Curtin University, WA.

P.K. Sarker, Associate Professor, School of Civil and Mechanical Engineering, Curtin University, WA

Name of Co-	Literature	Experimental	Data	Data	Discussion	Paper writing		
author	review	design/idea	collection	analysis				
J.C. Kuri					\checkmark			
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<u>Article 7</u>

M. N.N. Khan, M. M. A. Elahi, J. C. Kuri, P. K. Sarker and F. U. A. Shaikh. Performance of Alkali Activated Composites Containing Waste Glass Fine Aggregate against Acid Exposures. Advances in cement research, 2021 (Under Review).

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Name of Co-author	Literature review	Experimental design/idea	Data collection	Data analysis	Discussion	Paper writing
M.M.A. Elahi						
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P. K. Sarker		\checkmark		\checkmark	\checkmark	
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F. U. A. Shaikh		N		N	N	N
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(Signature)						

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1. M. N. N. Khan, J. C. Kuri and P. K. Sarker. Effect of waste glass powder as a partial precursor in ambient cured alkali activated fly ash and fly ash-GGBFS mortars. Journal of Building Engineering, 34 (2020) 101934.

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View Submission Author Status Correspondence Publishing Options Send E-mail	CLET-D-20-00255R2	Sustainable use of waste glass in alkali activated materials against H2SO4 and HCl acid attacks	Sep 20, 2021	Sep 28, 2021	Under Review

8. M. N.N. Khan, M. M. A. Elahi, J. C. Kuri, P. K. Sarker and F. U. A. Shaikh. Acid Resistance of Alkali Activated Composites Containing Waste Glass Fine Aggregate. Advances in cement research, 2021 (Under Review).

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