MINISTRY OF EDUCATION, RESEARCH, YOUTH AND SPORTS
TECHNICAL UNIVERSITY OF CIVIL ENGINEERING OF BUCHAREST
FACULTY OF CIVIL, AGRICULTURAL AND INDUSTRIAL

THESIS PAPER

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PhD THESIS

Development of Sustainable Geopolymer Mortar Incorporating Bottle Glass Wastes Nanoparticles

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Abstract

In the urban areas worldwide, energy saving, carbon dioxide (CO_2) emission levels reduction, and waste disposal due to excessive manufacturing of diverse consumer products became challenging. The industrial and agricultural wastes disposal remains the main environmental concern unless recycled in an eco-friendly way. The incorporation of these wastes into the concrete industries as substituting agents for some natural materials is strategized as one of the possible solutions. Only few percent of the million tons of annually generated glass bottle wastes are recycled. Recently, alkali-activated geopolymers with binders are introduced as environmental friendly construction materials alternative to the ordinary Portland cement (OPC) for lessening the CO_2emission and high energy consumption problems. The durability of concrete is the major concern in the construction sectors on globe. Concretes produced by recycling the agricultural and industrial wastes are shown to be environmental friendly with improved durability performance. However, the use of high amount of calcium-based materials into the concrete for improving the strength properties can affect negatively their durability performance in the aggressive environments. In this view, this study evaluated the effects of the bottles glass waste nanopowder (BGWNP) as ground blast furnace slag (GBFS) replacement in fly ash (FA)-based geopolymer mortars. The main aim was to examine the workability, strengths, microstructures, durability, and chemical properties of the proposed BGWNP blended geopolymers. Five batches of such alkali-activated mortars (AAMs) were prepared by replacing GBFS with 0, 5, 10, 15 and 20% of BGWNP with environmental friendly alkaline activator solution. Various analytical tests were performed to determine the flowability, viscosity, setting times, compressive, splitting and flexural strengths, porosity, drying shrinkage, and resistance to aggressive environments. Microstructures characteristics were evaluated using XRD, FESEM, EDX, FTIR, TGA and DTG measurements. Reuse of such wastes was found to remarkably improve the mechanical and durability traits of the produced geopolymers as well as solve the environmental and landfill problems. Geopolymer containing 5% of BGWNP as GBFS replacement showed improved compressive strength (above 16%) and durability improved with reduced porosity. The AAM composed of 10% BGWNP revealed reduced strength performance. Additionally, the replacement of GBFS by BGWNP was found to improve the durability performance in terms of reduced drying shrinkage and increased resistance to sulphuric acid, wearing and elevated temperatures. It is

established that the obtained geopolymers using BGWNP can offer much environmental benefits towards the lowering of global warming. Given that concrete still is the most used man-made material universally, this proposal would appreciably diminish the landfill needs for glass waste that is inappropriate for recycled glass production.

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CHAPTER 1

1.1 Introduction

Continued growth in the urban development projects worldwide has made ordinary Portland cement (OPC) increasingly in demand. As of now, OPC remains the principal binding agent in the concrete industry [1-3]. However, OPC is environmentally hostile because its manufacturing process releases high levels of carbon dioxide (CO_2) emission. For every ton of OPC production, one ton of CO_2 is produced (a typical ratio of 1:1). Thus OPC is classified as environmentally harmful binding material [4-6] as CO_2 is considered to be a significant factor in the production of greenhouse gases affecting climate change [7]. Due to industrial and urban development universally, the standards of living have undoubtedly improved. On the other hand, both industrial and domestic waste management has increasingly become a serious concern worldwide [7, 8]. Global collaboration for achieving greater efficiency in waste management, especially to recycle and repurpose the waste resources [9-12] has been noted. Such concerns have enforced the exploration of alternative options in terms of developing new environment friendly construction materials such as ‘green’ concrete as well as other products remanufactured from recycled wastes dumped in landfill [3, 13, 14].

Lately the alkali-activated mortars (AAMs) and concretes (AACs) have been introduced as cement-free materials. Generally, such mortars and concretes are prepared involving starting source materials rich in silicon (Si), aluminium (Al) and calcium (Ca) with alkali activation [15-17]. The starting resource materials include meta-kaolin (MK), fly ash (FA), palm oil fuel ash (POFA), ground blast furnace slag (GBFS) and ceramic wastes (WCP), etc., [18-20]. Compared to geopolymers, literature study on AAMs showed excellent properties such as fast setting time, curing at ambient temperatures [21], high early strength [22], high resistance to freezing-thawing cycles, good durability in aggressive environments [3], low CO_2 emission, cost and energy consumption [23, 24].

The alkaline activators solutions normally include sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) for the production of AAMs [25, 26]. Most of the research studies [22, 27, 28] revealed that the elevated concentration of NaOH (10-16 M) and high ratio of Na_2SiO_3 to NaOH (2.5) are preferred for the production of high performance AAMs. Na_2SiO_3 is known to impact negatively on the environment [29]. Besides additional cost, the high concentration of NaOH also effect negatively on the environment and remains hazardous to the workers [30]. High molarity of NaOH and enriched Na_2SiO_3 in the alkaline solution content are the major problems for the usage of AAMs as new construction materials. This is a serious concern for the environmental safety because AAM is a mineral based material that demands great amount of Na_2SiO_3 during synthesis. These deficiencies caused by alkaline solution limits the diversified applications of AAMs in the construction industry.

The inclusion of CaO from waste materials such as GBFS in alkali-activated mix led to enhance the strength even using low molarity (4 M) of NaOH [29, 31] as the Ca^{+2} can replace the Na^{+1} during the formulation of gels. The compatible nature of C-(A)-S-H and N-A-S-H gels has significant influence on the AAMs and alkaline solution activated alumina-silicate systems, wherein both products may be obtained [32].Despite the use of different source materials to prepare AAMs as binders, FA as industrial waste in particular, is immensely attractive for the synthesis of AAMs [33-35]. FA is a by-product of coal burning in thermal power plants to produce electricity which contains an ample quantity of amorphous alumina and silica [5].

Therefore, FA's chemical composition renders it a suitable resource material for producing alkali-activated binder. The properties of FA-based AAMs have been examined by several researchers. Due to their excellent durability properties, these AAMs are used as potential cementitious material. Many reports have acknowledged [3, 36, 37] similar engineering properties of AAMs making them favourable for various construction applications. Yet, several problems exist in using FA ($\geq$70%) based AAMs for slow setting time and low early compressive strength.

To overcome these drawbacks, one of the waste materials called ground blast furnace slag (GBFS) has been introduced for enhancing the FA based AAMs properties [28]. The major reaction products of alkali-activated cements for GBFS and FA are calcium silicate hydrate (C–S–H) and amorphous hydrated alkali alumina-silicate (N, C-A-S-H) [22], respectively. Alkali activated GBFS has high strength but issues related to rapid setting and insufficient workability along with high values of dry shrinkage, limits its applicability [3].Incorporation of GBFS in FA-based AAMs could enhance the workability, setting time and strength together with the reduction of alkaline activator solution demand. Otherwise, GBFS has negative effects on mortar's durability exposed to aggressive environments such as elevated temperatures, sulphuric acid and sulphate attacks. On top, the high content of CaO in GBFS is the main setback for the reduced resistance of mortar to aggressive environments. However, increasing the GBFS content in alkali-activated matrix can increase the cost, energy consumption and CO_2 emissions of AAMs mixtures.

Glasses are significant waste products that can potentially be used in concrete production a strategy which could be considered environment-friendly. Several million tons of glass bottles are discarded annually worldwide [38, 39]. Some of these wastes have already been recycled by glass manufacturers. Nevertheless, it is impossible to recycle all glass wastes due to the variation in colour, imperfections in glass, and processing costs.The potential for using glass in the production of concrete was first investigated some time ago [39, 40]. It has been established that pulverised glass wastes derived from bottles could contain significant amount of Al and Si apparently in non-crystalline form. These properties make glass waste a prospective pozzolanic or cement-like substance. Hence its usage as aggregates in cement production offers a suitable alternative for cement itself. However, it is worth noting that such use could change the characteristics of the finished products [41].

1.2 Study Background

Contemporary concrete production techniques place more emphasis on the inclusion of nanomaterials which offer enhanced features irrespective of whether it is fresh or cured. Most commonly used nanomaterials for performance enhancement of concretes and cement include nanoparticles, such as TiO_2, SiO_2, Al_2O_3, Fe_2O_3, carbon nanotubes/fibres and Nano silica owing to their pore-filling ability and favourable pozzolanic reaction [42-44].Meanwhile, steady increase in the demand of hybrid cement-like materials (extremely strong, durable, eco-friendly and sustainable) has been driven by the rapid development of infrastructure worldwide. The performance characteristics of even high-grade concrete can appreciably be enhanced by the addition of Nano-silica. In many cases, replacing concrete with as little as 6% nano-silica may achieve significant performance improvements [44].

It is the hydration process that is primarily modified by the introduction of nano-silica, which becomes effectively accelerated. This in turn leads to the generation of a greater quantity of calcium silicate hydrates through the reaction of nano-silica with calcium hydroxide within the concrete and hence the enhanced mechanical characteristics of the end product. The nano-silica impregnated concrete contains fewer calcium hydroxide crystals, which cause the formation of more compacted microstructures [45, 46]. Approximately 3% increase in the degree of pozzolanic reaction rate has been registered due to the addition of nano-silica in concrete [47]. Besides, a significant increase in the denseness, durability, tensile strength, compressive strength, bending strength and abrasive resistance has also been recorded in concrete impregnated with nano-silica [47, 48]. The permeability of concrete is also affected by the inclusion of nano-silica, with reduced permeability and capillary absorption [44]. Also, concrete containing nano-silica and ground GBFS disclosed enhanced splitting tensile strength and hydration speed [47].

1.3 Problem Statement

To enhance the AAMs' sustainability and durability performance in aggressive environments, we need to reduce the content of GBFS in mortar matrix. However, the reduction in amount of GBFS with increasing the content of FA affected negatively on strength performance of proposed mortars as well as increasing the porosity of AAMs' specimens. To solve this problem and produce high performance construction materials, developed new technologies need to apply. With such development and immense benefit of nanomaterials incorporated mortars, this study attempted to achieve high performance, durable and eco-friendly AAMs wherein a tiny amount of GBFS was replaced by nano-silica from bottle glass wastes.

The nano powder was prepared from glass bottles waste and characterized for its chemical and physical properties. The influence of bottle glass waste nano powder (BGWNP) inclusion in AAMs matrix as GBFS replacement was examined in terms of mortars workability, setting time, strength and microstructure. Durability of AAMs as a function of varied BGWNP content was also determined. The porosity, resistance to acid attack resistance, elevated temperatures, freezing-thawing cycles and abrasion from the synthesized BGWNP blended AAMs were evaluated. Results were discussed in terms of environmental benefits and sustainability of such AAMs.

1.4 Research Objectives

Based on the abovementioned problem statement the following objectives are set:

1. To characterize the chemical, physical and mineral properties of prepared BGWNP to producing sustainable alkali-activated materials.
2. To determine the optimum content of Nano glass powder and effect it on fresh and mechanical properties of synthesized alkali-activated mortars.
3. To evaluate the durability performance of prepared alkali-activated mortars.

1.5 Scope of the study

In this study, ternary binder containing high volume FA, GBFS and BGWNP were used to design high performance AAMs. Five batches made by replacing GBFS by 0, 5, 10, 15 and 20% of BGWNP in high volume FA-alkali-activated mortar. Low molarity of NaOH (2M) was considered and used to prepare the proposed mortars. For all AAMs, the solution modulus (SiO_2:Na_2O), binder to aggregate, alkaline solution to binder and sodium silicate content were fixed. Mortars properties such as flow ability, setting time, compressive strength, flexural strength, splitting tensile strength, porosity, ultrasonic pulse velocity, resistance to carbonation, sulfuric acid resistance, freezing-thawing cycles resistance, elevated temperatures resistance, scanning electron microscope (SEM), X-ray Diffraction (XRD), Energy Dispersive X-ray (EDX) are widely evaluated. All alkali-activated mortar specimens cured at same condition with 25±3 °C and relative humidity 75%. These new findings are validated and compared with art-of-the techniques exist in the literatures and similar results whenever available.

1.6 Research Significance

In general, using BGWNP in alkali-activated mortars can be led to enhance the specimens' strength and durable performance, saving landfill space and reduce the environmental problems. This research intends to generate new information on the use of nanotechnology and BGWNP blended alkali-activated mortars by means of systematic methods of specimen preparation from waste materials economically, appropriate and careful materials characterizations, and subsequent data analyses valuable for the progress of standard specifications of ternary blended alkali-activated towards diversified realistic applications. This generated knowledge is expected to provide to the advancement of environmental amiable and inexpensive alkali-activated mortar for broad array of usage in the construction sectors. This would be greatly advantageous for sustainable development of buildings, where wastes disposal problems towards the land filling can be avoided and minimized. The new findings of this research is believed to render a basis for further studies and better knowledge on the behaviour of a nano binder based alkali-activated obtainable from the waste material in a cheap and environmental affable manner.

1.7 Research Driving Questions

The research driving questions arising from the purpose are:

1. What the way to reduce GBFS content and not effect to lose the strength?
2. How can produce Nano powder high silica from glass bottle wastes?
3. Are the bottle glass wastes nano powder can improve the durability of mortar?

1.8 Novelty of the research

Utilizing nano powder from bottle glass wastes to replace the GBFS on high volume FA alkali-activated matrix, constituting a novel strategy with immeasurable environmental, technological and economic benefits. This study took an attempt to determine the role played by BGWNP when incorporated in FA-GBFS alkali-activated mortar as partial replacement with GBFS. Mortar specimens with varied predefined ratios of BGWNP and GBFS were designed. Performances of the prepared mortar specimens (fresh and hardened) were evaluated and compared with the control mix (0% BGWNP). As prepared mortar specimens were characterized using several tests to properties determine such as workability, durability, microstructure, and mechanical properties.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This chapter deals with the entire research program. Various laboratory experiments that were carried out to accomplish the proposed objectives. Laboratory studies were conducted to generate the data related to strength behaviour, durability performance, and microstructure patterns. It is worth mentioning that alkali-activated mortars (AAMs) being an emerging material does not have prescribed or standard procedures for the preparation and testing. However, standards for testing conventional Portland cement mortar and concrete were referred wherever applicable. Available research literatures in the alkali-activated area were also used as the basis of some procedures for the evaluation of AAMs and the standards for testing the synthesized specimens.

3.2 Experimental schedule

Figure 3.1 illustrates the entire experimental strategies to pursue this research. First phase deals with the materials characterizations which include the tests on physical, chemical and mineralogical properties of fly ash (FA), ground blast furnace slag (GBFS) and bottle glass waste nano powder (BGWNP). These characterizations were made to determine the mechanical and durable properties of ternary blended AAMs. Following the relevant standards or procedures outlined in the literature, various tests were carried out. Tests were conducted to determine both fresh and hardened properties of AAMs, where various mortar mixes containing FA and BGWNP as GBFS replacement were used. The minimum content of GBFS was limited to 10% level and at constant alkaline solution to binder ratio, binder to fine aggregate ration, sodium hydroxide (NH) molarity, sodium silicate (NS) to sodium hydroxide and alkaline activator solution modulus. Quantities such as flowability, initial and final setting times, compressive strength, flexural strength, splitting tensile strength, modulus of elasticity were evaluated to determine the fresh and hardened properties.

Tests were also performed to determine the microstructures and durability of proposed AAMs. The resistance of AAMs against sulphuric acid attack, elevated temperature, drying shrinkage, porosity and freezing-thawing cycles were also determined. In addition, microstructural evaluations were made to provide supplementary information regarding the influence of BGWNP on the strength properties of such AAMs. Such characterizations were performed using the thermogravimetry analysis (TGA), X-ray diffraction (XRD), Fourier transformed infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) and EDX.

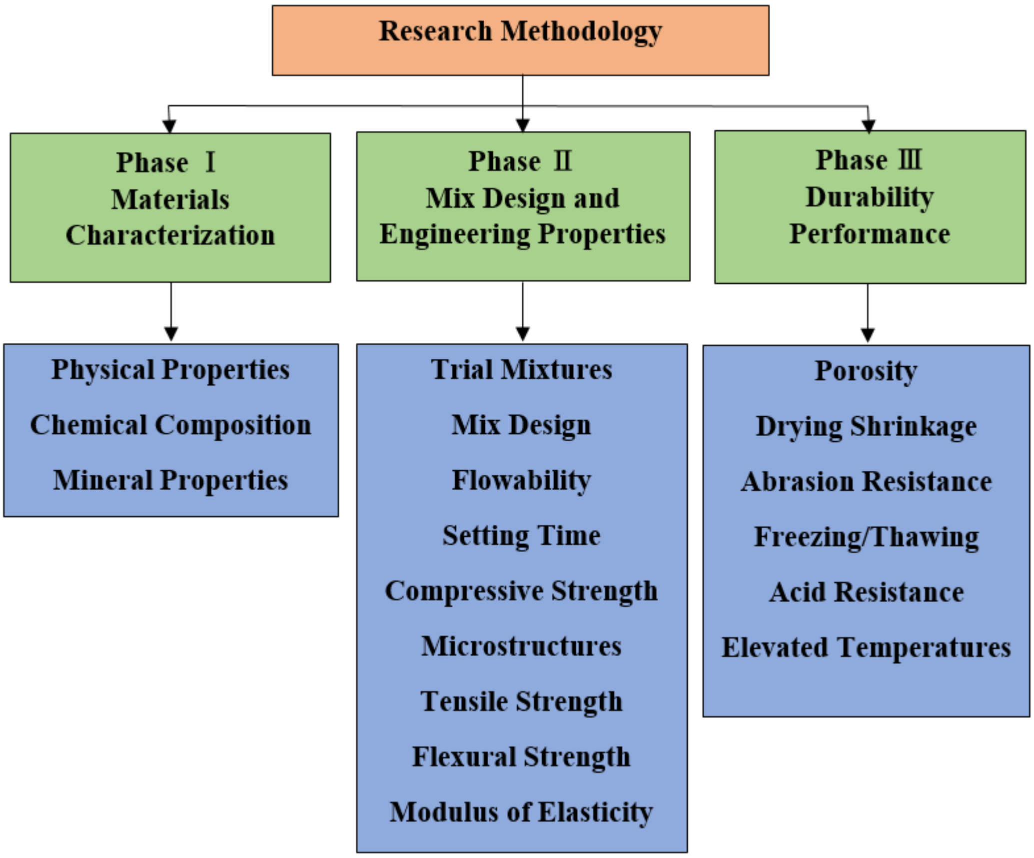


Figure 3-1 laboratory experiments

CHAPTER 4

Materials Characterization and Mix Design of Alkali-activated Mortar

4.1 Introduction

The formulation, design and characterization of alkali-activated mortar mixes were discussed in this chapter. Section 4.2 presents the results and discussion on chemical composite, particle size, morphology and thermal properties of fly ash (FA), ground blast furnace slag (GBFS) and Nano glass powder (BGWNP). In section 4.3, the trail mixes and control sample mix were studied. In the first category, the binary blend was prepared by mixing FA and GBFS with the ratio of 70:30 as control sample. The sodium hydroxide molarity, sodium silicate to sodium hydroxide and alkaline activator solution modulus were fixed to 2 M, 0.75 and 1.2, respectively. Next, the effects of optimum ratios of alkaline activator solution to binder (S:B), binder to fine aggregate (B:A) on the properties of AAMs are evaluated and the optimum ratios are selected for designing the mix. Section 4.4 emphasizes the mix design of ternary blended incorporating FA and Nano glass powder as GBFS replacement. These groups of material mixes were designed depending on the mass%. First group contained high volume FA (70%) blended with GBFS (30%) and considered as control sample in this study. The other groups were consisted of high volume FA (70%) blended with GBFS and Nano glass powder. GBFS was replaced by various levels of BGWNP to obtain the optimum mixture for high performance AAMs. Section 4.5 summarizes this chapter.

4.2 Materials Characterization

4.2.1 Chemical composition

Table 4.1 depicts the chemical composition and physical properties of BGWNP, GBFS and FA obtained using X-ray fluorescence spectroscopy. The primary oxide elements were found to be aluminium and silica, which are 83% in BGWNP, 41.7% in GBFS and 86% in FA. A significantly higher level (51.8%) of calcium oxide was found in GBFS compared to the BGWNP and FA.

Especially, the quantity of aluminium, silicate and calcium oxide influenced the synthesis of specimens considerably, through the formation of N, C-(A)-S-H gels as part of geopolymerisation process. Potassium oxide (K_2O) levels were lower than 1% for all three tested specimens. The sodium oxide (Na_2O) level in GBFS was appreciably higher (0.45%), whereas in BGWNP and FA it was 0.01% and 0.08%, respectively. Previous studies established that the activation of both the geopolymerisation and alkaline processes can be greatly influenced by K_2O and Na_2O. Besides, BGWNP, GBFS and FA exhibited reduced loss on ignition (LOI) values, which are in line with the standards set forth in ASTM C618.

Table 4.1: Chemical composition of FA, GBFS and BGWNP (weight %)

Material	FA	GBFS	BGWNP
SiO_2	57.20	30.8	69.14
Al_2O_3	28.8	10.9	13.86
Fe_2O_3	3.67	0.64	0.24
CaO	5.16	51.8	3.16
MgO	1.48	4.57	0.68
K_2O	0.94	0.36	0.01
Na_2O	0.08	0.45	0.01
SO_3	0.10	0.06	4.08
LOT	0.12	0.22	0.16

4.2.2 Physical properties

Table 2 demonstrates the physical properties of raw materials based alkali-activated binder included FA, GBFS and BGWNP. The particle size distributions of FA, GBFS and BGWNP obtained using particle size analyser. The calculated median particle size for FA, GBFS and BGWNP were 10000, 12800 and 80 nm, respectively. Furthermore, 100% of particles in FA and GBFS were below 45 µm size and 100% of BGWNP was passing 1 µm. Thus, these results fulfilled the pozzolanic requirement of ASTM C618 of 66 % passing 45 µm [298]. The FA presented grey colour compared to off-white and light-grey appearance of GBFS and BGWNP, respectively. The Brunauer, Emmett and Teller (BET) surface area of FA and GBFS were evaluated to determine the physical properties of the materials. Table 4.2 enlists their specific surface area and physical properties. For FA and GBFS raw materials, it was observed that the specific surface area of FA revealed the highest value of 18.1 m^2/g compared to GBFS which was estimated to be 13.6 m^2/g. The fine size and light specific gravity of FA have influenced on the surface area test which showed the highest value. However, the BGWNP presented 206 m2/g specific surface area and 1.02 specific gravity. Likewise, the nano particle of glass powder effect on surface area test and presented very high value compared to FA and GBFS.

Table 4.2: Physical properties of FA, GBFS and BGWNP

Materials	**Colour**	**Particle size (µm)**	**Specific surface area (m^2/g)**	**Specific gravity**
FA	Grey	10	18.2	2.20
GBFS	Off-whit	12.8	13.6	2.89
BGWNP	light-grey	0.08	206	1.02

4.2.3 X- Ray Diffraction Pattern

Figure 4.1 displays the XRD patterns of FA, GBFS and BGWNP. FA revealed sharp peaks at 2θ range of 16-30º which are assigned to the presence of crystalline alumina and silica. Moreover, other diffraction peaks were resulted from the occurrence of crystalline mullite and quartz phases. GBFS did not exhibit such peaks, confirming its amorphous characteristics. The presence of reactive calcium and silica substances is among the critical aspects of the formation of GBFS. In fact, presence of such reactive amorphous calcium and silica at high level make GBFS potentially ideal for AAMs production. The XRD pattern of BGWNP also showed its amorphous nature indicating that waste glass contained significant amount of reactive aluminium and silica. Furthermore, BGWNP disclosed weak crystalline quartz peaks at 27º and 29º.

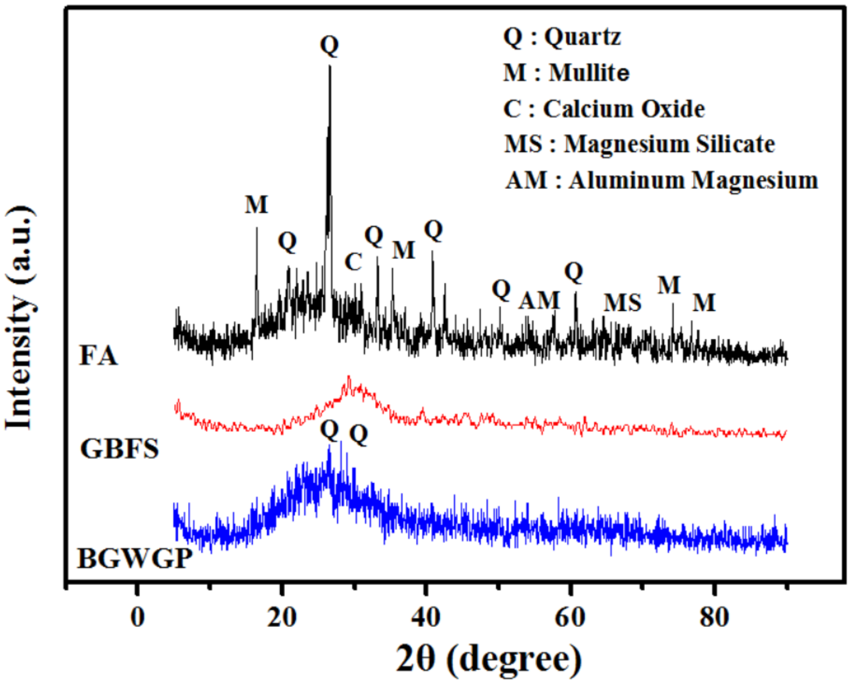
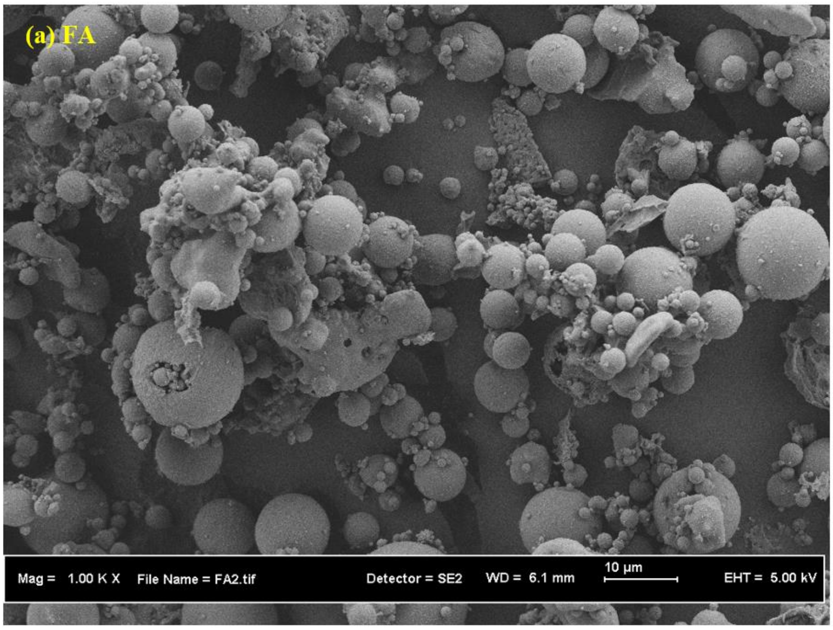


Figure 4-1: XRD patterns of FA, GBFS and BGWNP

4.2.4 SEM Analysis

Scanning electron microscopy (SEM) images of FA and GBFS are presented in Figure 4.2. It is evident that FA consisted of spherical particles (Figure 4.2a) with smooth surface. The GBFS (Figure 4.2b) comprised of irregular and angular particles which agreed with the previous report [299].



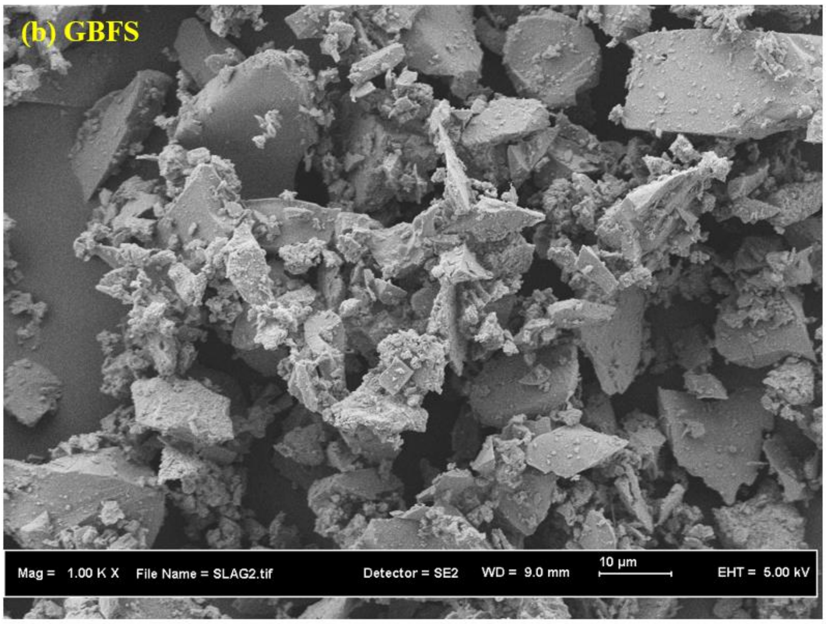


Figure 4-2: SEM micrograph of FA and GBFS

4.2.5 TEM Analysis

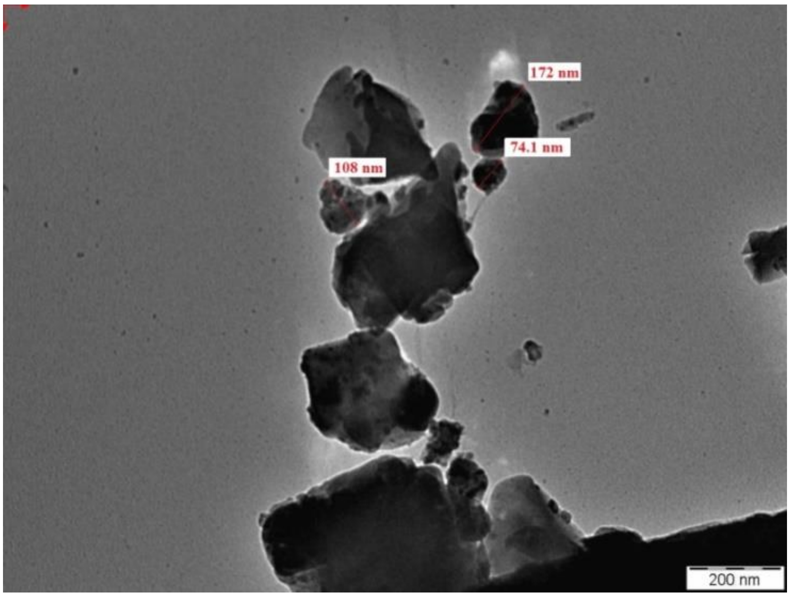


Figure 4.3 illustrates the TEM images of BGWNP which clearly showed the existence of ellipsoidal nanoparticles. Larger nanoparticles were also formed from the clustering of smaller ones, which was ascribed to the presence of high surface tension and surface energy of extremely refined nanoparticles. These tiny nanoparticles could produce a surface area which was extensive, thereby improved the nanoparticles' catalytic action. The mean size of the achieved nano-crystallite was 80 nm which agreed with those observed in nano-silica-based compound [33]. Varied super-elasticity has also been observed in BGWNP with similar constitution reported elsewhere [34].

4.3 Design of Trial Mixes

Binary binder content FA and GBFS with mass ratio of 70 and 30%, respectively was used to inspect the effect of alkaline activator solution to binder and binder to fine aggregate ratios. For all trial mixtures, the ratios of sodium silicate to sodium hydroxide, solution modulus and sodium hydroxide concentration were fixed to 0.75, 1.2 and 2.0, respectively. The influence of these factors was determined to select the optimum ratios to be used in the designed mixtures.

The high compressive strength after four weeks of curing age was the criteria to select the optimum mixture at all stages. The design of trial mixtures are described in the upcoming Sections 4.3.1 to 4.3.2.

4.3.1 Effect of alkaline solution to binder ratio

During the design of AAMs mix, the alkaline activator solution is the most important factor that affect the fresh, mechanical and durable properties of AAMs specimens. Six different ratios of alkaline solution to binder (S:B) were prepared to select the optimum one as delivered in Table 4.3. For all the mixtures, the molarity of NaOH, Na_2SiO_3: NaOH and binder to fine aggregate (B:A) were fixed to 2, 0.75 and 0.30, respectively. The properties of alkaline solution such as solution modulus (SiO_2:Na_2O) and total water to binder content (H_2O:B) are described in Table 4.3.

Table 4.3: Alkali-activated mortars mix design with various solution content, kg/m^3.

AAMs mix design							
Materials		MS_1	MS_2	MS_3	MS_4	MS_5	MS_6
Binder (B) kg/m^3	FA	420	420	420	420	420	420
	GBFS	180	180	180	180	180	180
Binder to aggregate content		0.30	0.30	0.30	0.30	0.30	0.30
Alkaline activated	S:B	0.30	0.35	0.40	0.45	0.50	0.55
	Solution	180	210	240	270	300	330
	NaOH	102.8	120	137.2	154.3	171.4	188.6

solution (S) kg/m^3	Na_2SiO_3	77.2	90	102.8	115.7	128.6	141.4
	H_2O	138.3	161.3	184.4	207.4	230.5	253.5
	Na_2O	18.9	22.1	25.3	28.4	31.6	34.7
SiO_2:Na_2O Mass%		1.2	1.2	1.2	1.2	1.2	1.2

Figure 4.4 illustrates the impact of alkaline activator solution to binder ratios on the compressive strength of free cement mortar. It was observed the increase content of solution from 0.30 to 0.35 and 0.40 led to enhance the geopolymerization process and increase the compressive strength from 36 MPa to 38 and 43 MPa, respectively. Unlike, the compressive strength value of mortars was decreased with increasing alkaline solution (more than 40%) to binder content. It implied that the compressive strength of free cement mortars was inversely proportional to the water to binder ratio (H_2O: B) which was similar to that of water to cement ratio in cement mortar. This finding was consistent with the previous reports of Sathonsaowaphak et al. [196] and Patankar et al. [300].

Moreover, the loss of compressive strength with increasing alkaline solution content implied the quantity of water present in the mix was much higher than the required as also confirmed by Chi [301]. It was reported that with increasing alkaline solution to binder ratio, more water inclusion results in higher capillary porosity. Meanwhile, less amorphous hydrated alkali-aluminosilicate gels were produced. Similarly, at alkaline solution to free cement binder ratio of 0.30, the mix was dry and difficult to compact properly, providing rough surface with reduced strength as reported by Mustafa et al. [302].

For alkaline solution to free cement binder ratio of 0.40 although the mix was very viscous and stiff but could achieve higher strength than other ratios. Again, this result was agreed to the report of Joshi and Kadu [303] and Abdullah et al. [304]. The effect of the Na_2O to Al_2O_3 ratio on the compressive strength of free cement mortars was also evaluated. The compressive strength revealed inversely proportionality to the ratio of Na_2O to Al_2O_3. Increasing alkaline solution also led to an increase in the Na_2O to Al_2O_3 ratio. However, the strength was reduced when the ratio was increased or reduced to 0.32 [305]. Zejak et al. [306] reported that the optimum Na_2O to Al_2O_3 ratio occurred in the range of 0.25 to 0.35 for maximum compressive strength of proposed mortars.

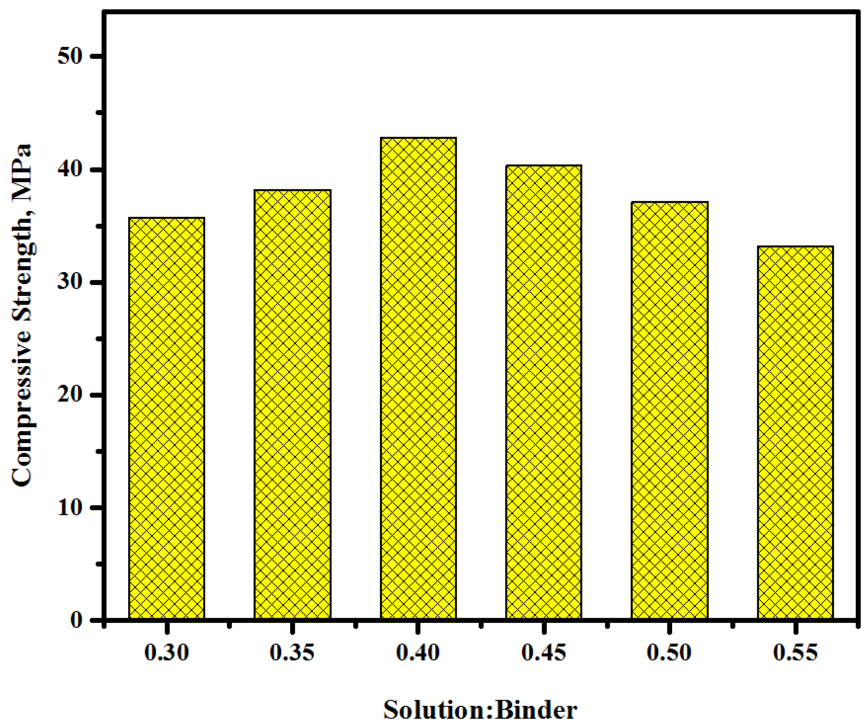


Figure 4-4: Impact of solution dosage on compressive strength development

4.3.2 Effect of binder to fine aggregate ratios

To evaluate the effect of binder to fine aggregate ratio (B: A) on the compressive strength performance of AAMs, six mixtures were prepared. The optimum compositions of NaOH, Na_2SiO_3: NaOH and (S: B) including 2 M, 0.75 and 0.40, respectively were used and kept fixed for all mixtures. Table 4.4 enlists the mix proportions of AAMs of various ratios of binder to river sand.

Table 4.4: Mix proportions of proposed mortars with various binder to aggregates ratios.

AAMs mix design							
Materials		MB_1	MB_2	MB_3	MB_4	MB_5	MB_6
Binder (B) kg/m^3	FA	420	420	420	420	420	420
	GBFS	180	180	180	180	180	180
Binder to aggregate content		0.30	0.60	1.0	1.50	2.0	2.5
Alkaline activated solution (S) kg/m^3	S:B	0.40	0.40	0.40	0.40	0.40	0.40
	Solution	240	240	240	240	240	240
	NaOH	137.2	137.2	137.2	137.2	137.2	137.2
	Na_2SiO_3	102.8	102.8	102.8	102.8	102.8	102.8
	H_2O	184.4	184.4	184.4	184.4	184.4	184.4
	Na_2O	25.3	25.3	25.3	25.3	25.3	25.3
SiO_2:Na_2O Mass%		1.2	1.2	1.2	1.2	1.2	1.2

The effect of binder to river sand content on development strength of proposed AAMs were studied. For this purpose, the compressive strength test was performed by taking three cubes from each set of binder to aggregate 0.30, 0.60, 1.0, 1.5, 2.0 and 2.5 respectively after 28 days. Figure 4.5 presents the compressive strength values for AAMs specimens prepared with various binder to fine aggregate content. The results illustrated that the compressive strength with high (2.5) and low (0.30) binder content display the low strength 42.8 and 43.1 MPa respectively compared to others alkali activated samples were prepared with 0.60, 1.0, 1.5 and 2.0 of (B:A).The results showed the compressive strength of specimens slightly trend to increase with rise the ratio of binder to fine aggregate to 0.60 and 1.0 and presented strength 47.9 and 56.2 MPa, respectively.

However, with rising the binder to fine aggregate ratio to 1.5 and 2.0 the strength dropped from 56.2 MPa to 49.3 and 45.3 MPa, respectively. It’s known the low ratio of (B: A) have effect on bond strength between the fine aggregate particles and show low strength. The produce of C-

S-H and C-A-S-H gels was reduced with increasing content of binder to 2.5 (B:A), with increase binder content the specific surface area was increased and led to increasing the alkali activated solution absorption which effects directly on dissolution of silicate and aluminium and shows low strength.This behaviour was similar to that of conventional mortar because the optimum proportion of fine aggregate yielded efficient binding by GP as reported by Joseph and Mathew [84] and Vasconcelos [307].

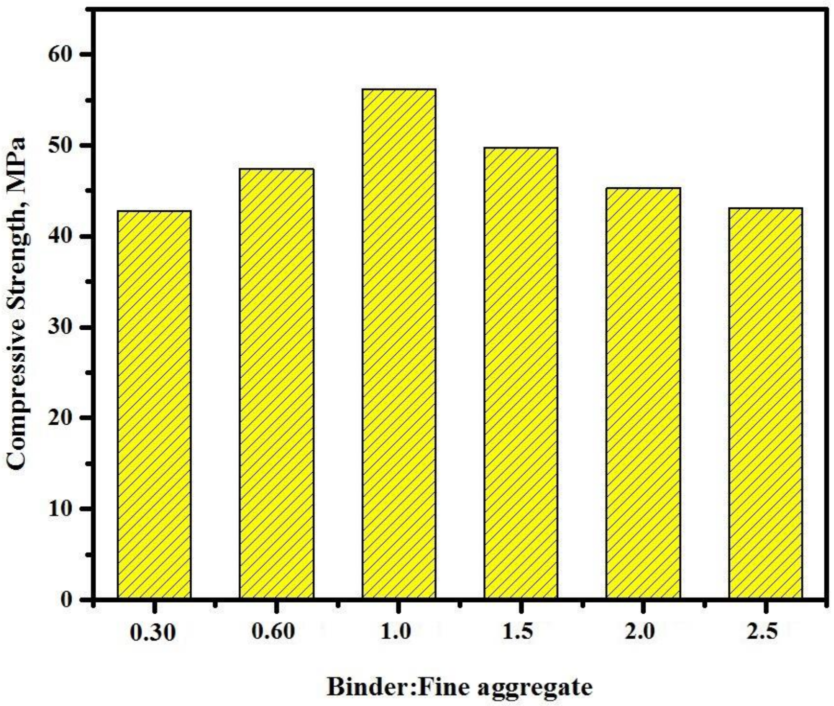


Figure 4-5: Impact of binder to fine aggregate content on strength development of AAMs

4.4 Mix design of AAMs

Based on trial mixes presented in section 4.3, the optimum ratios of alkaline activator solution to binder and binder to fine aggregate were selected as 0.40 and 1.0, respectively. These ratios were fixed for all mixes which was prepared to evaluate the effect of BGWNP on strength and durable performance of proposed mortars. Three types of waste materials were used to prepare the AAMs mix design. Ternary blended contents such as FA, GBFS and BGWNP were taken to determine the influence of nanomaterials on the geopolymerization process and engineering properties of AAMs.The minimum content of GBFS as a source of CaO was kept to a minimum of 10% in replacement process and maximum of 30%. Following the ASTM C109-109M standard, different mix proportions of GBFS: BGWNP were prepared in various ratios. In the first category, the blend was prepared by mixing FA and GBFS with the ratio of 70:30 as control sample. Next, the FA content was fixed to 70% by weight for all mixtures. The GBFS content was replaced by BGWNP with 5, 10, 15 and 20% by weight. Furthermore, at each level of replacement, the binder to fine aggregate (B: A), alkaline solution to binder (S: B), NH molarity, NS: NH ratio and modulus of solution (Ms) were fixed to 1.0, 0.40, 2, 0.75 and 1.2 by mass weight, respectively (Table 1). Testing was conducted in line with ASTM C579 standards. The tests consisted of (1) a 50-mm cube mould to test for compressive strength, (2) a prism (40×40×160) mm to test for flexural strength, and (3) a cylinder (length 150 mm, diameter 75 mm) to test for tensile strength. Prior to the moulds being cast, each one was coated internally with motor oil in order to facilitate easier demoulding. NS and NH solutions were mixed followed by cooling to ambient temperature before being used to avoid issues with heat produced during the mixing process.

The preparation of the Alkali-activated mortars (AAMs) involved the mixing of FA, GBFS and BGWNP for the duration of three minutes resulting in a uniform dry substance, which was then further mixed with fine aggregate for a period of four minutes. The resultant product was then activated by adding alkaline solution, and then blended in a machine for further five minutes at medium velocity. The final step in the process was the introduction of the resulting mortar into the moulds which was achieved by using two-layers pouring method. In this process, each layer being subject to vibration for a period of 15second in order to eliminate any air pockets within the mixture.

Once the casting process was completed, the AAMs were cured for 24 hours in an ambient atmosphere (temperature 24 ±1.5º C, relative humidity 75%) before demoulding process.

Table 4.5: Mix design of AAMs prepared with various ratios of BGWNP replacing GBFS.

Mix design formulation of alkali activated mortars						
Materials (mass,%)		$AAMs_1$	$AAMs_2$	$AAMs_3$	$AAMs_4$	$AAMs_5$
Binder (B)	FA	70	70	70	70	70
	GBFS	30	25	20	15	10

	BGWNP	0	5	10	15	20
Binder:Fine aggregate (B:A)		1.0	1.0	1.0	1.0	1.0
Solution:Binder (S:B)		0.40	0.40	0.40	0.40	0.40
Na_2SiO_3:NaOH		0.75	0.75	0.75	0.75	0.75
Sodium hydroxide (NaOH)	Molarity, M	2.0	2.0	2.0	2.0	2.0
	H_2O	92.6	92.6	92.6	92.6	92.6
	Na_2O	7.4	7.4	7.4	7.4	7.4
Sodium silicate (Na_2SiO_3)	H_2O	55.8	55.8	55.8	55.8	55.8
	Na_2O	14.7	14.7	14.7	14.7	14.7
	SiO_2	29.5	29.5	29.5	29.5	29.5
Total H_2O in alkaline solution		76.8	76.8	76.8	76.8	76.8
Modulus of solution (Ms) SiO_2:Na_2O		1.2	1.2	1.2	1.2	1.2

4.5 Summary

Based on findings the following conclusions can be drawn:

i. The chemical composition and physical properties of FA, GBFS and BGWNP satisfied the standard of ASTM C618.

ii. The results of PSA and TEM showed that the BGWNP achieved smaller size, lower specific gravity and highest specific area when compared with FA and GBFS.

iii. The FA presented crystalline structures compared to GBFS, BGWNP which were revealed amorphousness.

iv. The results of chemical composition showed that the BGWNP attained highest silicate content (69%) than other waste materials.

v. It is demonstrated that GBFS appeared to be the main resource of calcium in this experiment.

vi. From the trial mixes and compressive strength test, the results showed that the 0.40 and 1.0 of alkaline solution to binder and binder to fine aggregate ratios, respectively were the optimum one for the design mix.

vii. The content of calcium in design mix was significant where the main recourse of calcium (GBFS) was kept between 10 to 30%.

viii. The low sodium hydroxide molarity (2.0) and sodium silicate to sodium hydroxide (0.75) were adopted to prepare all AAMs mixtures.

ix. In AAMs mixtures, the GBFS was replaced by 5, 10, 15 and 20% of BGWNP to assess the effect of nanomaterials on workability, strength and durable performance of proposed mortars.

CHAPTER 5

Engineering Properties of High Performance Alkali-activated Mortars

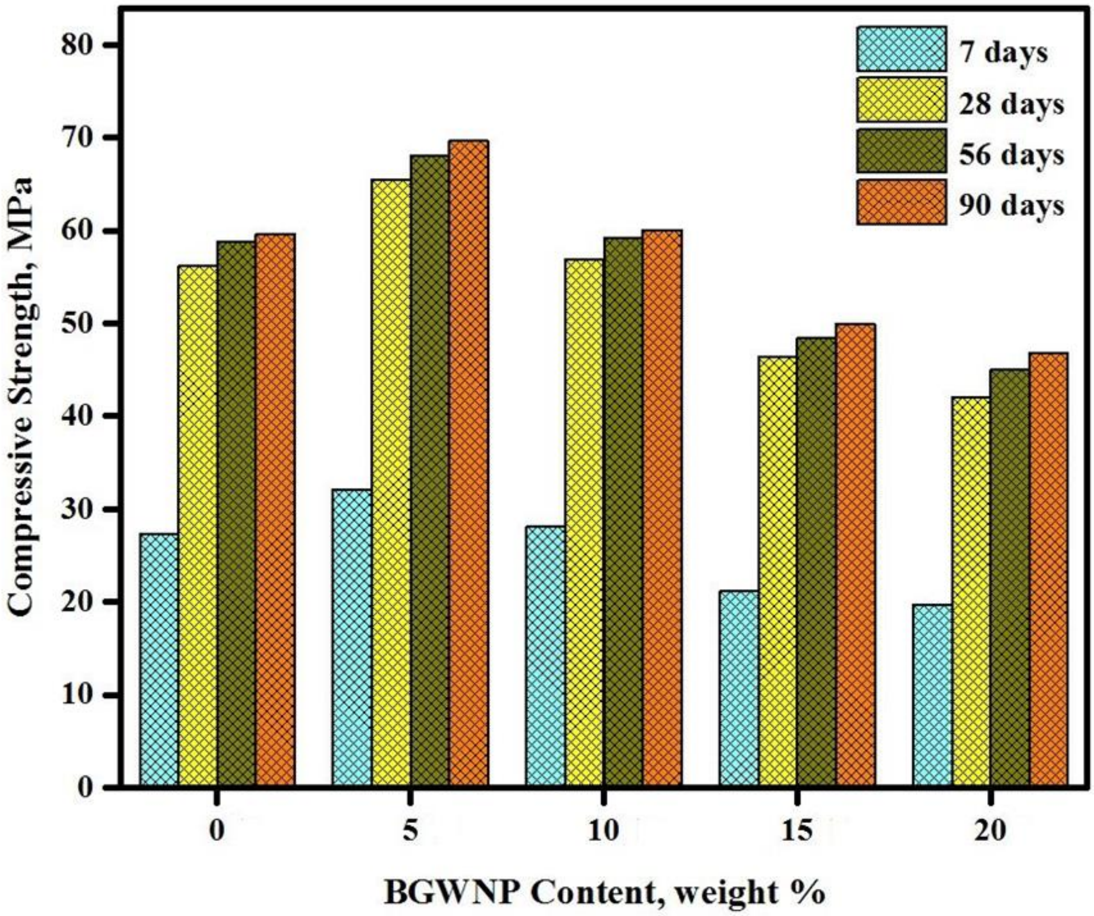
5.1 Introduction

In this chapter, the influence of Nano glass powder content as GBFS replacement on fly ash based alkali-activated mortars workability, mechanical and microstructures performance were evaluated. The flow, viscosity and setting times were considered to assess the performance of proposed mortars. To investigate the mechanical properties, several tests were used to evaluate the mortars performance included compressive strength, splitting tensile strength and flexural strength. The XRD, SEM, EDX, FTIR, TGA and DTG tests were adopted to characterize the microstructure of proposed mortars and effect of nano partials of glass powder to improve the morphology structure, produce the dense gels and reduce the total pores.

5.3.2 Compressive strength

Figure 5.5 displays the compressive strength of AAMs at ages of 7, 28, 56 and 90 days as a function of BGWNP content replacing GBFS. At early and late age, the compressive strength increased monotonically with the increase in BGWNP content from 0 to 5%. At 28 days of curing age, the compressive strength enhanced from 56.2 MPa to 65.5 MPa with increase in BGWNP content in AAMs matrix from 0 to 5%, respectively. However, the strength reduced to 42.1 MPa when the BGWNP content increased beyond 5% and reached to 20%. Likewise, the specimens prepared with 5% BGWNP achieved the highest strength compared to others for specimens tested at 56 and 90 days of curing age. With more benefits reported about the effects of nano-silica in cement- based materials, it is worthwhile that there are some contradicting results re: optimum percentage of nano-silica replacement.

It is fair to say that the gain in strength was mainly attributed to the method of nano-silica production and dispersion of BGWNP in cement-based materials. The main role of pozzolanic



reaction was strength development and reduction in pore size distribution. The increase in the compressive strength of mortar was noted up to 10% addition of nano-silica and reduced thereafter [46, 311]. Some of the studies [42, 44] reported that the addition of 4.0-6.0% of nano-silica by weight of FA could diminish the water absorption and result in denser structure, thereby improving compressive strength of mortar.

Figure 5-5: Effect of varying BGWNP content on compressive strength development of AAMs

CHAPTER 6

Durability Properties

1.1 Introduction

The results of hardened properties of respective AAMs are presented in this chapter. The results focused on the durability characteristics of AAMs system at different environmental conditions and compared to control sample. The comparisons of durability properties and behaviour include water absorption, resistance to sulphate and acid attacks, carbonation of proposed mortars, resistance to freezing-thawing cycles, abrasion resistance and the performance of AAMs at elevated temperatures. In addition, microstructural analysis was carried out in terms of SEM and XRD to support and better understanding the obtained results. With the discussions and results obtained from experimental test data, it clearly shows the effects of replacing GBFS by BGWNP used in the production of sustainable FA based alkali-activated mortars

6.2 Water absorption

Figure 6.1 depicts the water absorption of AAMs containing BGWNP at ages of 28, 56 and 90-days. The water absorption decreased with increasing BGWNP to just less than 10%. Water absorption property of AAMs was greatly influenced by the ratio of BGWNP replacing GBFS. The respective values obtained were 8.9, 9.6, 10.4 and 10.6 for 5, 10, 15 and 20% BGWNP content. It can be observed that the specimens containing 5 and 10% of BGWNP showed a packed pore structure which reduced water absorption compared to control sample (10.2%). However, with the increase in BGWNP level replacing more than 10% GBFS, water absorption augmented from 10.4 and 10.6% for BGWNP levels of 15 and 20%, respectively. Similar trend of results was also observed for the specimens tested at 56 and 90 days of curing age. The specimens containing 5% BGWNP showed the highest performance compared to other mixtures.

The reasons for such findings can be attributed as follows: as the BGWNP content increased from 0 to 5%, denser C-A-S-H gel was formed which enhanced the homogeneous strength with low water absorption. Also, the porosity of samples containing 5% BGWNP was reduced to about 12.7% compared to the control sample. This phenomenon could be attributed to the positive effect of BGWNP on the binder's hydration. Generally, the nucleation effect of BGWNP allows the formation of C–S–H phase which is no longer restricted to grain surface alone, thereby enhancing the degree of hydration of binder; also filling more pores by the newly generated C–S–H [313]. For this reason, the porosity of AAMs was found to decrease at first with the addition of BGWNP (5 and 10%). However, since the air content of AAMs increases with the addition of BGWNP, the porosity of BGWNP will increase again when the newly generated C–S–H is insufficient to compensate for the pores that are generated by the entrapped air in fresh mortar. Consequently, considering these two opposite processes, there is an optimal value of BGWNP amount at which the lowest porosity of AAMs can be obtained.

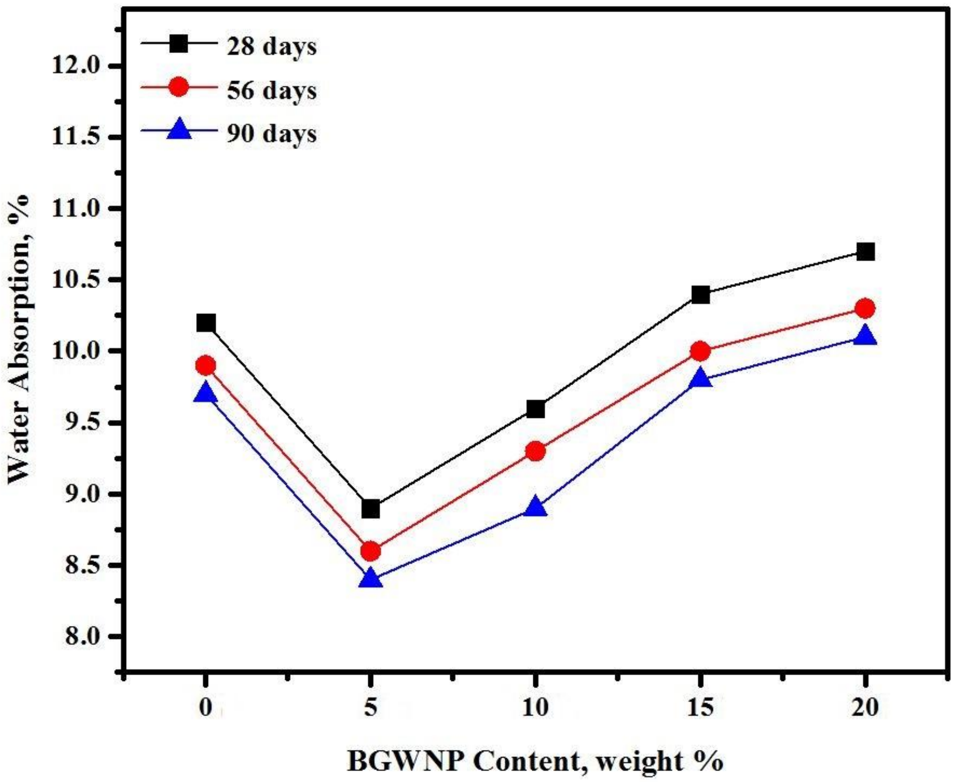


Figure 6-1: Water absorption of proposed AAMs

The relationships between the water absorption (WA) and the compressive strength (CS) of AAMs prepared with BGWNP as GBFS illustrated in Fig. 6.2. A reciprocal relation was exhibited between water absorption and compressive strength. At 28 days of curing age, the water absorption has dropped from 10.7 to 8.9% as the compressive strength of AAMs was increased from 42.1 to 65.5 MPa with the decrease in BGWNP replaced GBFS from 20% to 5%, respectively. Present results are similar to the one obtained by previous researchers [319, 320] where the water absorption was decreased with increasing compressive strength of AAMs. The linear regression method was applied to correlate the experimental data as shown in Eq. (6.1), with R^2 value of 0.86. This signified good confidence for the relationships.

$$WA = -0.0752\ CS + 13.837 \qquad \text{Eq. (1)}$$

Where $WA$the water absorption of AAM specimens is, CS is the compressive strength of the tested cube.

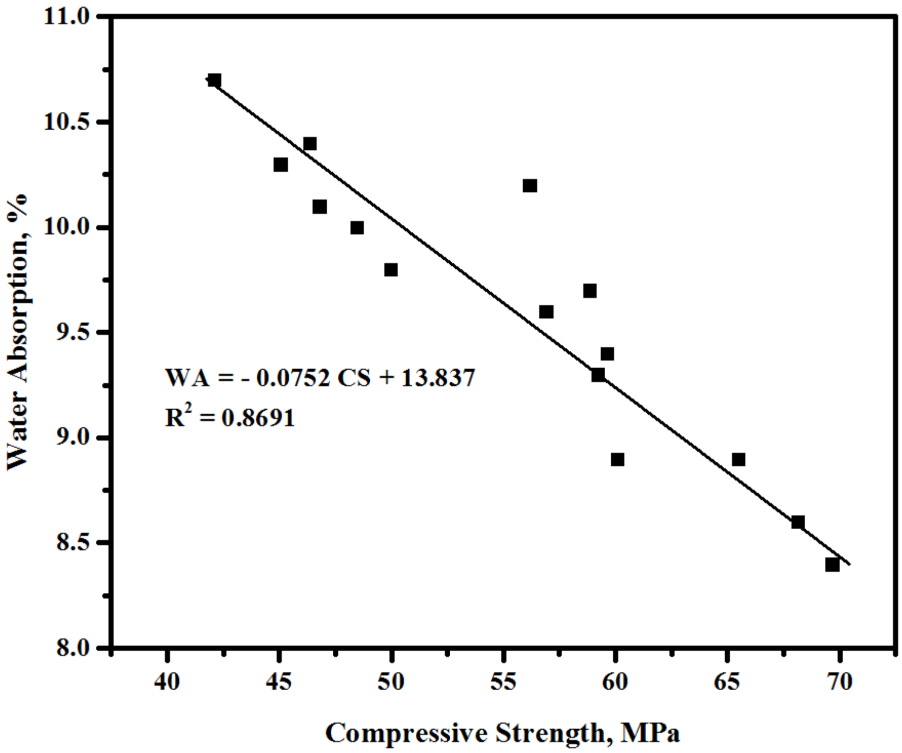


Figure 6-2: Correlation of water absorption and compressive strength

6.9 Summary

The influenced AAMs durability performance in term of water absorption, drying shrinkage, carbonation depth, abrasion resistance, resistance to freezing-thawing cycles, sulfuric acid resistance and resistance to elevated temperatures were evaluated in this chapter. Based on the findings the following conclusion were provided:

i. Inclusion BGWNP within 10% attributed to enhance the mortar durability by reduce the porosity and water absorption. Increase in the BGWNP content to 5% and 20% has enhanced the reaction process and produced denser C-S-H and C-A-S-H gel which further led to improve the microstructure of mortars with reduced voids.

ii. An inverse relationship was established among the compressive strength and porosity for the AAMs specimens. Inclusion of 5% of BGWNP as GBFS replacement enhanced the microstructures and compressive strength of the proposed mortar and reduced the porosity by 14.7% compared to the control sample.

iii. Addition nanomaterials (5%) in AAMs resulted to enhance the durability performance by reduce the carbonation depth. However, liner relationship was found between the porosity and carbonation depth.

iv. Inclusion BGWNP as GBFS replacement led to reduce the drying shrinkage and enhance the durability performance.

v. Proposed mortar prepared with 5% of BGWNP displayed the high performance against to freezing-thawing cycles. The reduction in porosity of AAMs has increased the resistance to freeze/thaw cycles.

vi. Direct relationship was observed between mortar resistance of wearing and compressive strength. The highest abrasion resistance achieved with specimens containing 5% BGWNP as GBFS replacement.

vii. Proposed mortars containing BGWNP showed very high resistance to sulphuric acid and attack. The specimens' resistance to acid attack trend to increase with increasing BGWNP, where minimum loss in strength of 7.3% was achieved for AAMs containing 20% of BGWNP in the blend.

viii. The detailed microstructures analyses (using XRD) of the BGWNP incorporated mortars displayed the formation of less amount of free Portlandite and gypsum, reduction in the porosity, improvement in the durability performance against the sulphuric attack.

ix. Increased in the BGWNP content has enhanced the elevated temperature resistance of AAMs in term of residual strength and weight loss.

x. Results of XRD and SEM explained the thermal stability of AAMs containing high level of BGWNP (20%) when exposed to heat.

xi. A practical advantage of studying surface discolouration of fired-AAMs in the preliminary assessment of damage caused by fire hazards, so that intensity of fire can be comprehended.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

7.1 Introduction

The Conclusions and recommendations sections sum up the key points of the findings, achievements, and contribution of the research, and also bring acknowledgement for further research related to this filed. This chapter presents the general conclusions on the findings of this study and the contributions made. These conclusions are emerged based on the results and discussion enclosed in Chapters 4, 5 and 6.

7.2 Conclusion Based on Characterizations of Constituent Materials

The physical characteristics, chemical composition, morphology and microstructure of raw materials based alkali-activated binder included FA, GBFS and BGWNP were studied, as well as the characteristics of the filler (river sand) and alkaline activator solution (sodium hydroxide and sodium hydroxide). As well as, the trial mix design prepared with various ratios of alkaline solution to binder and binder to fine aggregate content were evaluated in aim to optimize the alkaline solution and fine aggregate content. Based on the results can be highlighted as the following:

i. Waste materials based alkali-activated binder included FA and GBFS were utilized without any laboratory treatment. BGWNP was prepared by crushed the collected bottle glass then followed by grinding to achieve the nano size (less than 100 um).

ii. The median of particle for FA, GBFS and BGWNP wERE estimated to be 10, 12.8 and 0.08 µm, with specific surface area 18.1, 13.6 and 206 m^2/g. Thus, these size distributions of ingredients as blend for alkali-activated binder fulfilled the pozzolanic requirement of ASTM C618 of 66% passing 45 µm.

iii. Unlike to FA, X-ray diffraction analysis of GBFS and BGWNP were found to be completely amorphous without any crystalline phases.

iv. The chemical composition of GBFS revealed a rich calcium and silica content, compared to FA and BGWNP. The main element in FA and BGWNP were silica and aluminium. Keep GBFS content between 10 to 30% contributed to modification of the CaO/SiO_2 and SiO_2/Al_2O_3 ratio through binary blending.

7.3 Conclusion Based on Characterizations Trial Mixes and Mortar Mix Design

i. Binary binder contents including FA and GBFS with mass ratio of 70 and 30%, respectively, were utilised to inspect the effect of alkaline activator solution to binder and binder to fine aggregate on the alkali-activated blends. The optimum ratio was used in the designed mixtures. The high compressive strength after 28 days was the criteria to select the optimum mixture at all stages.

ii. Depended on trial mixtures results, the alkaline activator solution to binder, binder to fine aggregate, sodium hydroxide concentration, sodium silicate to sodium hydroxide and solution modulus were fixed to 0.40, 1.0, 2.0, 0.75 and 1.20, respectively in preparation of proposed mortars.

iii. To evaluate the effect of nanomaterials on workability, strength and durable properties of proposed mortars, ternary blended were prepared by incorporating FA with GBFS replaced with 0, 5, 10, 15 and 20% of BGWNP.

7.4 Conclusion on the Effect of BGWNP on Workability Performance

i. It was observed the flow diameter of tested mortars slightly trend to decrease with increasing BGWNP as GBFS replacement.

ii. The initial and final setting times took longer for mixtures containing BGWNP as replacement of GBFS.

7.5 Conclusion on the Effect of BGWNP on the Strength Properties

i. The prepared friendly AAMs with low molarity of sodium hydroxide and amount of sodium silicate which could achieve a compressive strength above 56.2 MPa in at 28 days.

ii. The achieved compressive strength, flexural strength, splitting tensile strength and modulus of elasticity were optimum for AAMs containing 5% BGWNP as replacement of GBFS. However, increased replacement levels up to 10% negatively affected the AAMs strength.

iii. Inclusion 5% of BGWNP as GBFS led to improve the splitting tensile strength compared to control sample.

iv. BGWNP showed positive effect on the development of flexural strength and modulus elasticity after 28 days compared to control sample.

v. XRD, FESEM, EDS, FTIR and TGA analyses showed the enhancement in microstructures and C-S-H gel formation of AAMs containing less than 10% BGWNP.

7.6 Conclusions on the Assessment of Durability Properties

i. Inclusion of BGWNP in AAMs matrix was highly effective to enhance the durability and to reduce water absorption capacity. AAMs prepared with 5% and 10% of BGWNP showed better performance than the control sample.

ii. The drying shrinkage values trend to decrease with increasing BGWNP. Controlling the GBFS content in alkali-activated mixtures shown positive effect on durability performance by reduction the drying shrinkage values.

iii. The loss in compressive strengths, UPV, mass and surface textures deterioration of the AAMs exposed to the sulfuric acid attack were decreased with the increasing BGWNP content as GBFS replacement. The formulation of gypsums were reduced by reducing the GBFS content in alkali-activated matrix.

iv. AAMs specimens prepared with 5% BGWNP as GBFS replacement presented high as well as increasing resistance to abrasion in early and late age.

v. Increase in the BGWNP content in the alkali-activated matrix could enhance the resistance to elevated temperatures. An increase in the BGWNP content could significantly reduce the deteriorated of tested specimens compared to control sample.

vi. The high resistance to freezing-thawing cycles was observed with specimens containing 5% of BGWNP a GBFS replacement.

vii. Replacement of GBFS by 5% of BGWNP enhanced the durability performance, reduced the porosities and carbonation depth compared to the control sample.

viii. Replacement of GBFS by BGWNP can be regarded as environment-friendly where re-used of glass bottle waste could diminish the CO_2 emission, save energy and reduce the total electricity and fuel consumption, thus leading to cost reduction and enhanced sustainability.

ix. Recycling the glass bottles waste could reduce the landfill problems, energy savings and minimize the demand of natural materials in construction industry.

7.7 Research Contributions

In short, the main constraint of wide range application of AAMs in the construction industry is related to the environmental issues, cost problems of high molarity alkaline activator solution and low durability to aggressive environments. This research could partly solve those problems by reducing the calcium level in the new alkali-activated ternary blend where a decrease in the GBFS content was advocated. This research extended knowledge of application of friendly alkali-activated mortar preparation as new construction materials through the evaluation of the impact of alkaline solution with low molarity on various properties of proposed mortar. However, significant contributions of the research towards providing new information on the behaviour of the ternary blended alkali-activated mortars which were not achieved before. This study shed some light on the effect of BGWNP as GBFS replacement on the workability, strength, durability properties.

The complementary utilization of BGWNP and GBFS in alkali-activated matrix to cover the low level of sodium on dissolution and reaction in geopolymerization process is the novelty of this study. Through systematic preparation of new alkali-activated blends and their thorough characterizations it was demonstrated that such ternary blends can offer the flexibility to achieve good mechanical properties and durability in order to meet the required specifications of construction industries. It is established that the exploitation of this new alkali-activated blend could promote both economic and environmental sustainability of the construction industry.

7.8 Recommendations

Based on the findings and conclusion of the study, here are several recommendations to be considered:

i. The effect of BGWNP as GBFS replacement on sustainability and life cycle assessment of alkali-activated mortars.

ii. It is important to develop friendly alkali-activated concrete with desired properties needed for the precast industry.

iii. The effect of BGWNP on the self-compacting of alkali-activated concrete need in-depth investigation.

iv. Evaluation the feasibility of using proposed mortars as repair materials in different environments.