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Laboratory evaluation of alkali-activated mortars modified with nanosilica from glass bottle wastes

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ABSTRACT

Saving energy, reducing greenhouse emissions, and the eco-friendly disposal of waste materials produced during manufacturing processes are significant challenges in urban communities around the world. Every year, over a million tons of glass bottles are disposed of. In fact, very few are recycled. In the present research, Nanopowder (NGP) (a waste product of glass bottles) was prepared by placing slag (GBFS) in fly ash (FA)-based alkali-activated mortars (AAMs). The key objective of the study was to measure the compressive strength performance and microstructure of the NGP blended AAMs. It was revealed that reusing these waste products enhanced the strength and microstructure properties of the AAMs that were produced. In the AAMs whereby 5% NGP was used to replace GBFS, compressive strength (above 16%), microstructure properties and durability were all enhanced with lowered water absorption. However, the strength performance of the mortar made up of 10% NGP was lower. Thus, the researcher concluded that there were clear environmental advantages of using the proposed AAMs consisting of NGP, which could significantly minimize global warming. As concrete remains the most used man-made material around the world, the reuse of AAMs could thus significantly reduce landfill requirements for glass waste that cannot be recycled for further glass production.

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1. Introduction

Global collaboration for achieving greater efficiency in waste management, especially to recycle and repurpose the waste resources 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 [1–4]. In free-cement concrete industry, alkali-activated binders based by-product and agricultural wastes are introduced as environmental friendly materials with high strength performance [5]. It is thus a viable alternative for replacing traditional concrete. Amorphous calcium (alumina) silicate hydrate (C–(A) –S–H) is the significant by-product of alkali-activated binder reactions with GBFS and FA [6]. Although the strength of GBFS based AAMs is impressive, there are major problems with its rapid setting and insufficient workability. Moreover, its applicability is also restricted due to its high level of drying shrinkage [7] and lower durability performance when exposed to attacks by sulphate and sulphuric acid. GBFS has a high CaO content, which is the primary drawback of using reduced-resistance mortar in harsher environments [8]. However, the applicability, settings time and workability can be improved by using GBFS in FA-based AAMs, whilst simultaneously lowering the alkaline activator solution demand. On the other hand, replacing GBFS content by high amount of FA led to reduce the compressive strength as well as more porous microstructure [9].

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The waste products in glass may be used in the production of concrete in an environmentally-friendly manner. Every year, millions of tons of glass bottles are thrown away around the world [10,11]. Although some waste glasses are recycled, recycling all types of glass is impossible for various reasons, including colour variations, glass imperfections and the costs of recycling. The use of glass waste in concrete production has been a topic of interest for a while [11,12]. Pulverised glass wastes obtained from bottles may contain large quantities of aluminosilicate in non-crystalline form, which ultimately render glass waste a potential pozzolanic or cement-like substance. Thus, it may serve as a viable alternative to cement. Nonetheless, using glass waste may change the properties of the finished products [13].

Modern concrete production methods tend to include nanomaterials that possessive enhanced features. The nanomaterials most often used to improve concrete performance include carbon nanotubes/fibres, nanoparticles (e.g. TiO_2, Al_2O_3, Fe_2O_3), and Nano silica. This is because they can fill pores and generate favourable pozzolanic reactions [14–16]. Concrete containing nano-silica possess less calcium hydroxide crystals, resulting in more compacted microstructures [17,18]. The addition of nano-silica in concrete has been found to increase the pozzolanic reaction rate by around 3% [19], and to cause significant increases in denseness, tensile strength, durability, bending strength, compressive strength, and abrasive resistance [19,20]. Furthermore, the use of nano-silica has also been found to lower permeability and capillary absorption in concrete [19]. Concrete consisting of nano-silica and ground GBFS also has a more favourable hydration speed and splitting tensile strength [19].

Given the developments outlined above, in addition to the significant advantages of using waste nanomaterials incorporated in concrete production, the present research aims to create high performance, durable and environmentally-friendly mortars by replacing a small amount of GBFS with NGP. Nano powder was taken from wastes glass bottle, after which its chemical composition and physical traits were assessed. The study focused on exploring the strength and microstructure of the mortar when NGPs were used to replace GBFS in AAMs.

2. Methodology

2.1. Materials

In the present study, the ternary alkali-activated blend was created using source the following source materials: FA, GBFS and NGP. FA is a fundamental source of aluminium–silicate and was obtained from local supplier. It was not treated in any way before use. GBFS, which served as a cement-free material, was obtained from north Malaysia (Ipoh). The supplier pulverised it to the required particle size before transporting it for the experiment. GBFS served as the primary source of calcium and silicate. After collecting the waste glass bottles from Skudai (Malaysia), the researcher then cleaned them with tap water to get rid of contaminants and then crushed by crusher machine. Crushed glass was sieved through 600 μm to separate large glass particles. Subsequently, Los Angeles Abrasion Machine with 25 kg capacity was utilized to grind the sieved glass for 3 h in order to get medium particle size of 25 μm using 16 ϕ 40 mm stainless balls. Later, the resultant powder was heated in an oven at 110 °C (±5) for 60 min and again ground for 7 h using ball mill machine to achieve optimum distribution of nanoparticles.

In preparation the proposed mortar, sodium hydroxide (NH) and sodium silicate (NS) were mixed to prepare the alkaline activator solution (AAS), which activated the aluminosilicate. Pure NH (98%) and Na_2SiO_3 were bought from the Quality Reagent Chemical (QREC) Asia company in Malaysia. A NH solution with low molarity (2 M) was chosen to prepare the AAS, and a Na_2SiO_3 to NH ratio of 0.75 was used for all samples. This ensured that the environmental impacts of NH and NS were reduced as much as possible. To prepare the NH solution, pellets were dissolved in tap water and kept for 24 h at room temperature. The solution consisted of SiO_2 (29.5 wt%), Na_2O (14.70 wt%) and H_2O (55.80 wt%). Subsequently, to create the final alkaline solution, NS was added to the NH solution. To produce all of the mortar samples, naturally-occurring siliceous river sand was employed as a fine aggregate. To purify the sand, it was washed in water according to ASTM C117 standards. After this, it was placed in the oven for 24 h at 60 °C to dry. The sand was then graded to ensure that it met the ASTM C33-33M specification.

2.2. Mix design and test procedure

In line with the ASTM C109 standard, ternary blended included FA, GBFS and NGP were mixed in different ratios. In preparation the proposed mortar, the first blend was created using a mixture of FA and GBFS to the weight ratio of 70 to 30, respectively. This serves as the control sample. After this, the FA content was set at 70% for all mixtures. To replace the GBFS, NGP was used in increasing amounts (5, 10, 15 and 20%) by weight. Moreover, at each replacement stage, the binder to fine aggregate (B: A) was set at 1.0, the AAS to binder (AAS: B) at 0.40, NH molarity at 2, the NS: NH ratio at 0.75 and modulus of solution (Ms) at 1.2 according to their mass weights (see Table 1). When performing the tests, ASTM C579 standards were followed. The tools used in the tests included: a 50 mm cube mould for measuring compressive strength. Before casting each mould, they were each coated internally using motor oil. The purpose of this was to make demolding easier. After mixing the NS and NH solutions, they were cooled to the atmosphere temperature before being tested. This ensures that any heat-related issues caused throughout the mixing process are avoided.

The NGP, BGFS and FA were mixed for three minutes to prepare a uniform, dry substance for AAM preparation. A fine aggregate was then mixed into the substance for four minutes. Subsequently, an alkaline solution was mixed into the substance and machine-blended for five minutes at a medium speed. Finally, the mortar produced in the process is placed into a mould via a two-layers pouring method. Each layer was then subjected to vibration for 15 s to ensure that the mixture contained no air pockets. Following the completion of the casting process, AAMs were cured at atmospheric temperature (24 ± 1.5 °C) and relative humidity of 75% for 24 h. After this, they were demoulded.

A number of tests were performed to examine the impacts that Nanoparticles from glass waste have AAMs properties. In the concrete production industry, exploring the impacts that additional materials have on strength and performance is imperative. This is because such properties are vital indicators of sustainability. Compressive strength tests were performed to measure the strength performance of AAMs impregnated with NGP to replace GBFS. Moreover, several tests were performed to investigate microstructure, including TGA, FTIR, SEM and XRD. This offers a deeper insight into the mixture's strength properties.

After 28 days of curing, the center of each AAM was removed and pulverized, after which the powder's microstructure was examined by conducting XRD, SEM, EDx, FTIR, TGA and DTG analyses. The Match3 and MDI Jade software were employed in the X-ray diffraction test to identify the distribution of the alkali-activated mortar samples. The results of the scanned specimens were analysed across a 2Θ range of 5-90°, with 0.02 steps and a scan speed of 0.5 s/step. The newly-produced AAMs were then placed on brass stub holders and dried for five minutes via infrared

Table 1
Proposed alkali-activated mortar mix design prepared with different ratio of NGP as GBFS replacement.

Materials (mass,%)		AAMs mixtures				
		AAMs-M_1	AAMs-M_2	AAMs-M_3	AAMs-M_4	AAMs-M_5
Binder (B)	GBFS	30	25	20	15	10
	NGP	0	5	10	15	20
	FA	70	70	70	70	70
B:A		1.0	1.0	1.0	1.0	1.0
AAS:B		0.40	0.40	0.40	0.40	0.40
NS:NH		0.75	0.75	0.75	0.75	0.75
NH	M	2	2	2	2	2
	H_2O	92.6	92.6	92.6	92.6	92.6
	Na_2O	7.4	7.4	7.4	7.4	7.4
NS	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		76.8	76.8	76.8	76.8	76.8
AAS-Ms		1.2	1.2	1.2	1.2	1.2

radiation. The specimens were then coated with gold using a blazer sputter coater. Finally, a 20 kV with 1000× magnification was used to identify the resultant patterns.

3. Results and discussion

3.1. Raw materials characterization

X-ray fluorescence spectroscopy (XRF) was adopted to find the chemical composition of binder' materials included FA, GBFS and NGP. Silica and aluminium were identified as the predominant oxide elements, constituting 86% in FA, 83% in NGP, 41.7% in GBFS. In BGFS, the calcium oxide was found to be much more prominent than (51.8%) than in NGP and FA. The amounts of aluminium, silicate and calcium oxide had a significant impact of the synthesis of specimens because they facilitated the formation of dense gels during the geopolymerization process. In all three test samples, potassium oxide (K_2O) levels were below 1%. Moreover, the level of sodium oxide (Na_2O) detected in GBFS was discernibly greater (0.45%) than in NGP (0.01%) and FA (0.08%). Prior research has found that K_2O and Na_2O largely influence alkaline processes and geopolymerization. Additionally, a reduced loss on ignition (LOI) was revealed in the NGP, GBFS and FA specimens, and this is in line with ASTM C618 standards. FA had a calculated median particle size of 10000 nm, whilst for GBFS it was 12800 nm and for NGP 80 nm. All particles present in FA and GBFS were less than 45 μm and all those in BGWNP exceeded1 μm in size. Whilst the FA appeared to be grey, GBFS gave an off-white tone and NGP had a light-grey appearance.

3.2. Compressive strength development (CS)

In Fig. 1, the proposed mortar strength after 28 days impregnated with NGP is presented. It appears that there is a monotonical increase in compressive strength when the NGP content increases from 0 to 5%. Moreover, the CS rose from 56 MPa to 65 MPa when the NGP content was heightened from 0 to 5%. Nonetheless, once the NGP content exceeds 5%, the compressive strength gradually decreases, reaching 42.1 MPa with a 20% increase in NGP content. Although there are many advantages of using nano-silica in concrete production, prior studies demonstrate some contradicting results in terms of the optimum percentage of nano-silica replacement. It seems that the increase in strength was primarily due to the technique used to produce nano-silica and disperse NGP. The pozzolanic reaction predominantly serves to enhance strength and to reduce the pore sizes. The study showed that concrete's compressive strength continues with up to 8% addition of nano-

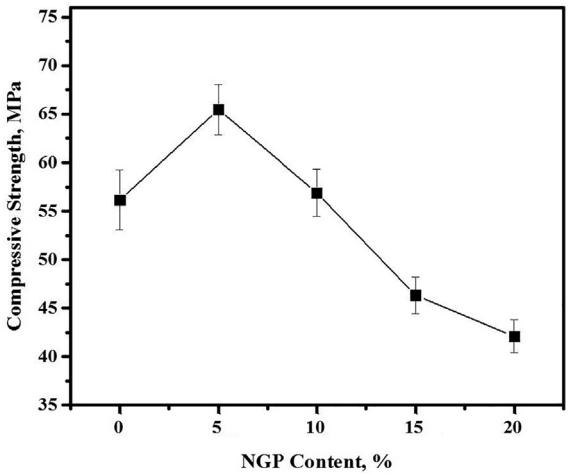

Fig. 1. Impact of NGP level as GBFS replacement on CS development at 28 days of age.

SiO_2, after which it decreases [18,21]. Several researchers [14,16] have found that adding 4% to 6% of nano- SiO_2 by weight of waste of fly ash may reduce the amount of water absorbed and cause the structure to be denser. Ultimately, this would enhance the compressive strength of concrete. However, the reduction on compressive strength for the specimens prepared with greater than 10% NGP could be attributed to high water demand. Consequently, it produced an adverse effect on the hydration process.

3.3. XRD pattern of AAMs

In Fig. 2, the XRD patterns, as well as the AAM's crystalline structure after 28 days are presented. Between 20° and 35°, an amorphous hallow can be seen, which suggests that AAM gel is present. The XRD patterns show that the NGP took effect in the AAMs between 23.9° and 34.1° as the intensity of albite ($Na0.95Ca0.05Al1.05Si_2.95O_8$) and gismondine ($CaAl_2Si_2O_8{\cdot}4(H_2O)$) reached their peak. Nonetheless, the intensity of quartz (SiO_2) peak (36°) was reduced when NGP was added to the proposed mortar. The increase in gismondine and albite and the decreases SiO_2 peaks indicate that denser gels have been produced. This plays a significant role in facilitating the hydration and geopolymerization processes. Compared to the zero NGP speciemns, When the amount of GBFS replaced by NGP was between 5%, the albite and gismondine peaks were found to have the

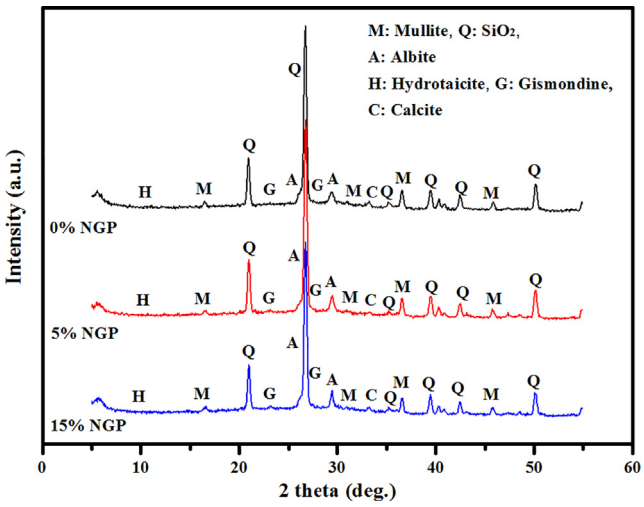

Fig. 2. XRD patterns of AAMs prepared with various NGP content.

highest intensity. However, when the quantity is increased 15%, a significant effect is exerted on the geopolymerization process and less dense gels are produced in the AAMs. This, in turn, reduces the strength of the substance.

3.4. SEM and EDx analysis

To investigate the effects of using NGP instead of GBFS in AAM mixtures, microstructural image analyses were performed. The scanning electronic microscopy (SEM) images and energy-dispersive X-ray (EDx) spectra for the new mortars are presented in Figs. 3 and 4. The samples containing 5% of NGP were found to have a high-performance structure, fewer pores and less non-reacted particles (Fig. 3b) compared to control specimens (Fig. 3a). The FA partial reaction can be seen in the SEM images. Moreover, the number of unreacted FA spheres still present in the mortar mixtures is significant. As well as amorphous reaction products, there were also partial crystalline structures measuring 150–300 nm in diameter. Additionally, several needle-shaped crystals were identified on the surfaces and areas surrounding the FA particles, with some such crystals being covered in a layer of dense amorphous gel. Jang et al. [22] also highlighted the existence of these crystals in their study. On the whole, the SEM images showed the AAMs to contain crystals, unreacted FA and a reacted gel phase. Increasing the amount of NGP added (15%) caused a rise in the number of non-reacted particles, which had low-density structures (see Fig. 3c).

In the EDx spectra shown in Fig. 4, it is evident that the 1.15 ratio CaO to SiO_2 was high in the samples containing 5% NGP. However, the CaO in the mixture appeared to decrease further when more NGP was added. On the other hand, the CaO to SiO_2 ratio was lower in the sample containing 15% NGP (0.64). Moreover, when the NGP level was increased from 5% to 15%, there was also an increase in the SiO_2 to Al_2O_3 ratio from 1.74 to 2.55. In the spec-

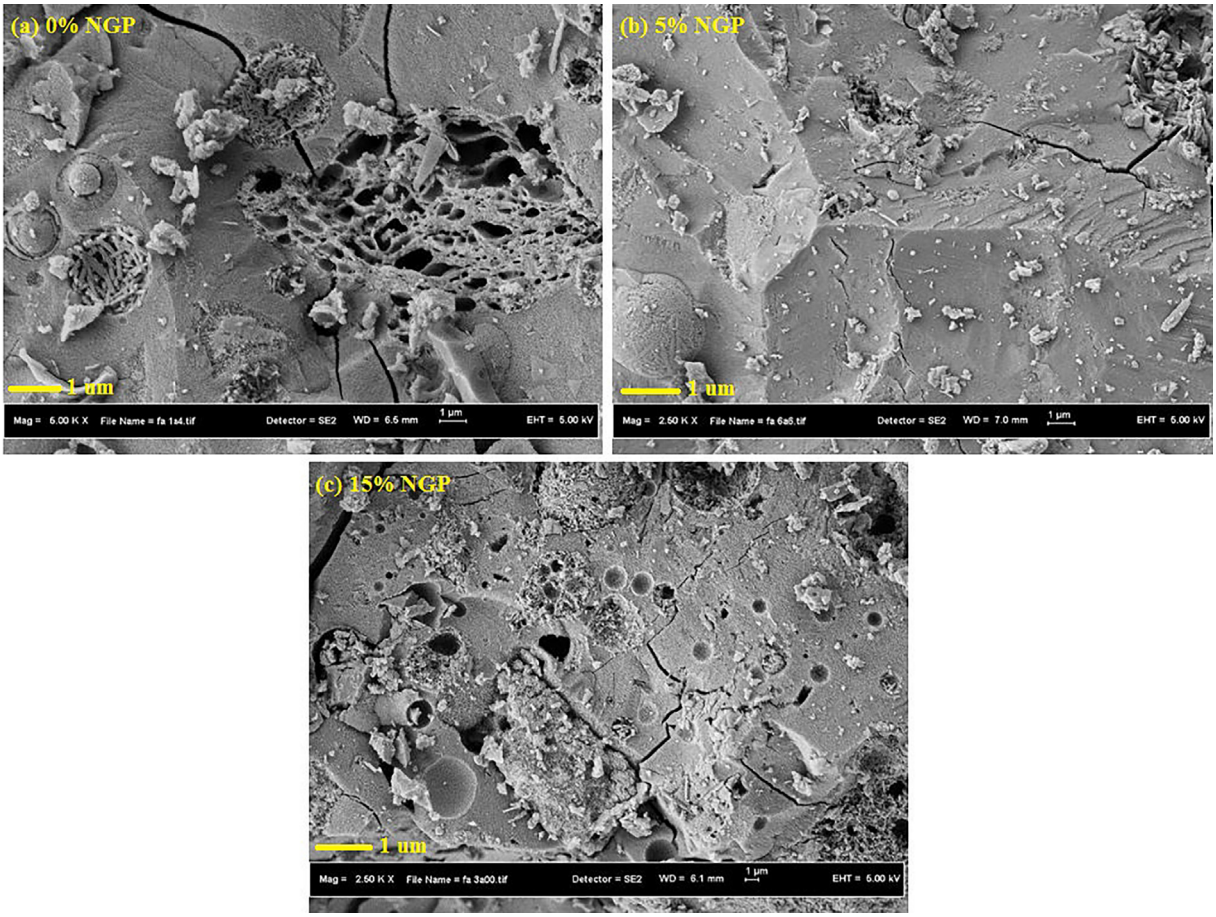

Fig. 3. Effect of NGP on AAMs surface morphology (a) 0% NGP (b) 5% NGP (c) 15% NGP.

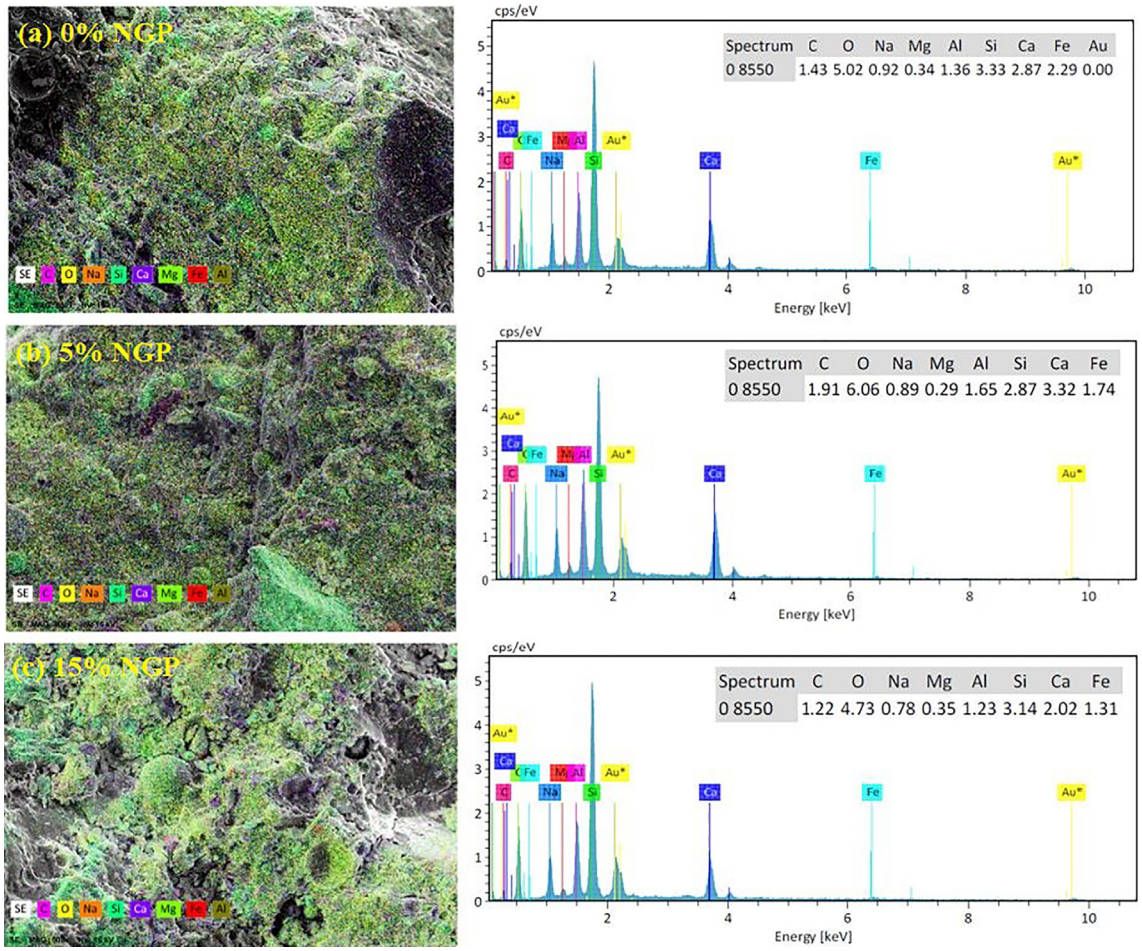

Fig. 4. AAMs EDx map (a) 0% NGP (b) 5% NGP (c) 15% NGP.

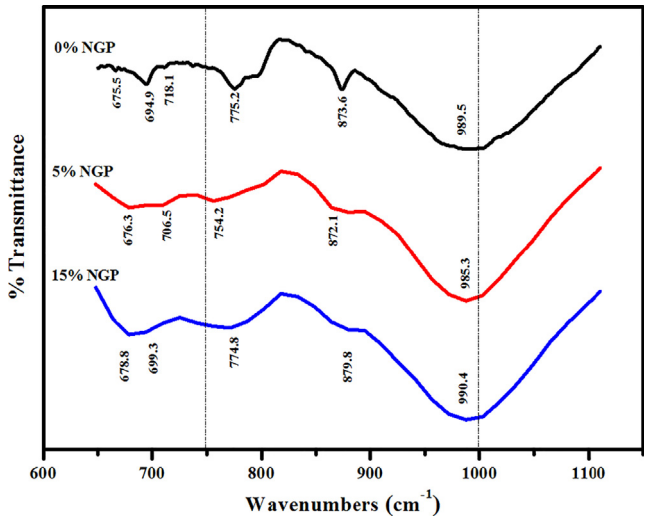

Fig. 5. Effect of NGP content on AAMs' FTIR spectra.

imens containing 5% NGP, the primary difference was that relatively low SiO_2/Al_2O_3 levels were revealed in the EDx spectrums. This could suggest that the higher Al ions have been replaced in the dense gels chain. Moreover, the decrease in CS found when NGP content was increased from 5% to 15% may be caused by lower levels of Al_2O_3 and CaO due to increased SiO_2. Ultimately, this

Table 2
Chemical compositions of FA, GBFS and BGWNP.

Material	FA	GBFS	BGWNP
Silicon oxide (SiO_2)	57.20	30.8	69.14
Aluminium oxide (Al_2O_3)	28.8	10.9	13.86
Iron(III) oxide (Fe_2O_3)	3.67	0.64	0.24
Calcium oxide (CaO)	5.16	51.8	3.16
Magnesium oxide (MgO)	1.48	4.57	0.68
Potassium oxide (K_2O)	0.94	0.36	0.01
Sodium oxide (Na_2O)	0.08	0.45	0.01
Sulfur trioxide (SO_3)	0.10	0.06	4.08
loss on ignition (LOI)	0.12	0.22	0.16

caused fewer gels to be produced in comparison to the 5% NGP. The high stability of AAMs containing 5% NGP was attributed to the presence of high amount of C-S-H gel and low percentage of Ca $(OH)_2$. This clearly suggests the benefit of NGP in enhancing the microstructure properties and increasing the strength performance of proposed AAMs.

3.5. FTIR spectra analysis

To investigate the formation of products in the reaction, as well as the extent of geopolymerization in different AAM solutions, FTIR measurements were assessed. Chemical analyses were carried out to identify functional groups based on bonding vibrations. By doing this, the Al-O and Si-O reaction zones in mortar mixtures could be determined by the FTIR (see Fig. 5). In these mixtures, the com-

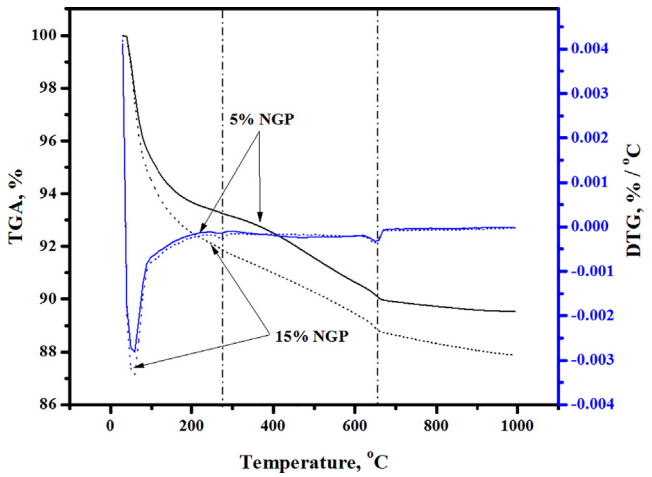

Fig. 6. AAMs; TGA thermal analysis of 5% and 15% NGP.

pressive strength increased as the minerals dissolved, which occurs when alkaline activators are inserted into the base materials. This causes hydroxylation to occur, which facilitates the release of A and ultimately, it results in –OH ions in the alkali attaching to create an Al–O–Al bond. By doing this, the weak bonds that enabled negatively charged Al to be released in the IV fold coordination are replaced. Lastly, by adding Ca, it was possible to achieve a balanced charge. The Ca reacted more favourably than Na [23]. Furthermore, as presented in Table 2, Ca solubility was higher in the GBFS samples containing more CaO than FA (Table 2). The amount of Ca dissolved is determined by how much GBFS is contained in the mixture. This has a direct impact on the overall compressive strength of the mixture. During the process of polycondensation, the unit oligomer of (–Si–O–Al) may take the form of chains, sheets or 3D-frameworks. This allows the product to harden [24].

The key objective of the present research was to improve the durability, eco-friendliness and strength performance of AAMs that use NGP instead of GBFS. When increasing the NGP content from 0 to 5%, the strength of the substance after 28 days was significantly improved (from 56.2 to 65.5 MPa). On the other hand, increasing the levels of NGP to 10, 15 and 20% reduced the strength to 56.9, 46.4 and 42.1 MPa, respectively. These changes are presented in Fig. 5. Subsequently, the Si–O–Al ring in 0% NGP (989.5 cm^{-1}) transformed to 985.3 and 987.9 cm^{-1} for the new mortar when the levels of NGP used were 5 and 10% respectively. There was an evident reduction in the Si–O–Al band frequency which caused an increase in C(N)-A-S-H gel produced. Due to this, the AAM structures at 5 and 10% NGP had high homogenous structures and greater silicate re-organization than the original 0% NGP sample. It was found that, when the NGP level is increased from 15 to 20%, reduces the compressive strength of the mixture and causes an increase in the band frequency reading to 990.4 and 995.4 cm^{-1}. Moreover, when changing the NGP level from 0% to 5%, the Si–O-Si bending modes decreased from 775.2 cm^{-1} to 754.2 cm^{-1}. This suggests an increase in the production of C-S-H gel. As the molecular mass of attached atoms increased, there was a reduction in the vibration frequency. Therefore, the soluble Ca produced by the GBFS displaces the Si atoms in the Si–O bonds, thus causing the vibrational frequency to be lowered. An increase in both the vibrational frequency (Si-O-Si) and the SiO_2/Al_2O_3 ratio was caused by the use of NGP [25].

The results showed that, in Ca-based free cement mortar, the hydroxylation of akermanite and gehlenite produces condensation, which leads to the formation of C_3AS_3 and Ca-disiloxonate hydrate [26]. A further factor indicating structural reorganization is that, in the specimens containing 0, 5 and 10% FA binders, the AlO_4 vibration band shifted from 873.6 cm^{-1} to 872.1 and 874.8 cm^{-1} respectively. Thus, structural changes were caused by increased levels of NGP in the AAMs. These changes may be caused by the increase in dense gels that are produced when the quantity of added nano-silica is higher. It is important to note that the geopolymerization rate was decelerated due to these changes, and the mechanical strength of the AAMs was significantly reduced.

3.6. TGA and DTG thermal analysis

In Fig. 6, the TGA and DTG curves for the new AAM mixtures incorporating 5 and 15% of NGP can be seen. The purpose of performing TGA thermal analyses was to identify the percentage of weight lost in the AAM mixtures. The samples containing 5% NGP lost less weight (10.47%) than the sample with 15% of NGP, the latter of which lost over 12.12% of its original weight and had high stability. Additionally, in the 5% NGP sample, there was a higher percentage of dense gel in AAM (9.63%) than in the 15% NGP sample (7.82%). The researcher also calculated the calcium hydroxide percentage and found that the 15% sample contained the highest percentage (5.73%), with the 5% sample only containing 4.53%. The 5% sample was found to have high stability, and this is due to high levels of dense gel and low levels of Portlandite being present in the mixture. Thus, the advantages of using NGP to improve microstructure and strength performance are clear to see. It was also asserted by Singh et al. [27] that, when Nano powder is used, the products of hydration cause the bulk paste matrix to become denser, whilst also improving the mechanical properties and durability of concrete.

4. Conclusions

The present paper investigated the viability of using Nano powder (a product found in waste glass material) instead of GBFS in the production of AAMs. In-depth experimental analyses were performed, and enabled the following conclusions to be made: (1) the elasticity modulus and compressive strength were highest in the mixture containing 5% NGP instead of GBFS. Nonetheless, the strength of the AAMs was reduced when the NGP levels were increased to 10%. (2) Based on the microstructure test analyses, it was determined that the microstructures and dense gels (N,C-(A)-S-H) formation in proposed mortars with under 10% NGP were improved. (3) The process of replacing GBFS with NGP is environment-friendly and the reuse of waste glass may generate a significant reduction in harmful CO_2 emission. It can also save energy, as well as lower electricity and fuel consumption. This thus makes it a more cost-effective and sustainable option. (4) The many issues relating to landfill, energy-saving and demand for natural materials that are prominent in the construction industry can be largely reduced through the recycling of waste glass.

CRediT authorship contribution statement

Hussein K. Hamzah: Methodology. **Zahraa Hussein Joudah:** Writing - review & editing. **Dan Paul Georgescu:** Supervision. **Nur Hafizah A. Khalid:** Supervision. **Ghasan Fahim Huseien:** Writing - original draft, Methodology.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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