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Comparison of Microstructural Properties of One- and Two-Part Fly Ash Geopolymer Concretes

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Abstract. Geopolymerisation is a chemical process involving reacting raw aluminosilicate minerals with alkali activators such as sodium hydroxide and potassium hydroxide to produce a geopolymer binder. In most cases, anhydrous activator is typically made by dilution with water to form a liquid activator before being blended with other raw components such as sand and coarse aggregates. This traditional approach, also known as two-part mixing, can be hazardous due to the corrosive nature of the liquid activator, making it difficult to mix huge volumes of concrete. In recent years, a one-part mix or “just add water” geopolymer concrete has been proposed as a simpler mixing method to minimise mixing time and improve the perception of geopolymer concretes. Similar to the preparation of typical Portland cement concretes, a one-part mix is performed in which all of the dry ingredients, including the solid activator, are initially combined together before water is eventually added to the mix. As a result, the aim of the research is to compare these two geopolymer concrete types, as well as how these mixing processes affect the mechanical strength and microstructural properties of fly ash geopolymer concretes. This study also compares the effects of the activators used, which included sodium hydroxide (NaOH) and potassium hydroxide (KOH). Microstructural investigation was performed through Fourier Transform Infrared (FTIR) and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDX). It was observed that one-part geopolymer concrete using NaOH as activator shows higher mechanical strength. The fly ash geopolymer binder study reveals that sodium aluminosilicate hydrate (N-A-S-H) gels and potassium aluminosilicate hydrate (K-A-S-H) gels are the binding gels formed in one-part and two-part mixing methods at different alkali activators with the formation of N-A-S-H gels is faster with NaOH activator than with KOH. It may be inferred that one-part geopolymer concretes perform better than two-part geopolymer concrete mixtures, and that NaOH activated concretes exhibit more desired characteristics than KOH activated concretes. It can be concluded that one-part geopolymer concretes perform better compared to two-part mix of geopolymer concretes and NaOH-activated concretes gives desirable properties compared than KOH.

INTRODUCTION

Geopolymer is created by the alkaline activation of low-cost materials or industrial waste, such as fly ash, slag, and rice husk. Fly ash geopolymer is a cement-free building material that not only protects the environment (Gartner and Hirao, 2015) and (Flatt, Roussel and Cheeseman, 2012), but also often outperforms OPC in terms of performance and durability (Bakri *et al.*, 2013; Ismail *et al.*, 2013; Aiken *et al.*, 2017). Corrosive hydroxides such as sodium hydroxide (NaOH) are frequently utilised in the manufacture of these geopolymer concretes. Traditionally, these concretes are mixed using anhydrous pallets of NaOH diluted with raw precursors and other materials such as gravel and sand (Singh *et al.*, 2008) and (Kumaravel and Girija, 2013). This technique is referred to as two-part mixing, and it has been successfully used on a large scale (Glasby *et al.*, 2015). However, it is time-consuming and requires additional safety measures owing to the activator solution's corrosive nature. Meanwhile, one-part fly ash geopolymer concrete, or simply "just add water," is made by mixing all of the dry components together, including the solid anhydrous activator, and then adding water (Bong *et al.*, 2021) (Nematollahi *et al.*, 2020). Duxson and Provis (2008) and Thomas *et al.* (2016) started the development of one-part geopolymer many years ago. At the moment, a one-part geopolymers remain commercially viable in comparison to two-part geopolymers (Glasby *et al.*, 2015).

The type of activator solution used to activate the fly ash is critical to the reaction progression. When fly ash is activated with solutions of NaOH and KOH in the presence of sodium silicate and potassium silicate, it has no crystalline phase other than the crystalline of fly ash. However, when activated with NaOH solution, the fly ash will include some hydroxysodalite in addition to the minerals found in the fly ash. When fly ash is activated with KOH solution, potassium carbonate and potassium bicarbonate are produced (Palomo, Grutzeck and Blanco, 1999). In comparison to the KOH 18M solution, the NaOH 12M solution provides more strength and quicker activation. The residual hydroxide ion concentration in KOH when 18M is used weakens the alkali cement (Palomo, Grutzeck and Blanco, 1999). The presence of an alkaline solution has a significant effect on the microstructure of a two-part fly ash geopolymer. In comparison to the KOH solution, the NaOH solution has a greater solubility of silica and alumina at the same concentration.

The microstructure of the two-part fly ash geopolymer is denser than that of OPC, resulting in much less chloride transport and porosity. The denser microstructure results from a greater reactivity with fly ash as a result of the alkaline activator's higher alkali concentration. García-Lodeiro, Palomo and Fernández-Jiménez (2007) also discovered that one-part and two-part fly ash geopolymers performed better than OPC concretes. Additionally, the performance of one- and two-part geopolymers is dependent on the amount of silica in the alkaline activator (Palomo, Grutzeck and Blanco, 1999). Criado, Fernández-Jiménez and Palomo (2007) concur, stating that a product with a greater silica content will have a higher mechanical strength.

The difference between a one-part and two-part fly ash geopolymer lies in the amount of silica and aluminium present, as well as the rate of release. Hajimohammadi, Provis and Van Deventer (2011) demonstrated that a one-part affluent-silica geopolymer paste with a low silica concentration produced a crystalline phase with a greater silica content than a pure geothermal silica geopolymer paste. Additionally, one of the advantages of geopolymer-based concrete over OPC concrete is its resilience to heat. Both one-part and two-part fly ash geopolymers outperform OPC concretes in terms of heat resistance (Sarker, Kelly and Yao, 2014, Sarker and McBeath, 2015).

The purpose of this study is to compare the mechanical characteristics and microstructures of one-part and two-part fly ash geopolymer concretes as a function of activator. In order to make a one-part fly ash geopolymer concrete, anhydrous activator (NaOH and KOH) pallets are mixed together with other ingredients. For a two-part mix, the alkali solution must be prepared and left for equilibrium for 24 hours. Compressive strength testing, functional group determination testing, and microstructural observation will be performed on both techniques to determine the strength development of the fly ash geopolymer concrete after 7, 14, and 28 days. However, the microstructures of one- and two-part geopolymers are not extensively characterized. As a result, this study examines the mechanical and microstructural characteristics of fly ash geopolymer concrete as a function of the mixing technique, namely one-part and two-part mixing.

METHOD AND MATERIAL

Fly ash

The fly ash utilised in this study was provided by Sejangkat Power Plan, which is located in Kuching, Sarawak. As indicated in Table 1, the mineral composition of Class F fly ash is composed of the following elements:

Table 1: Chemical composition of fly ash

Element	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	K ₂ O	Others	LOI
Composition	4.91	55.90	21.80	6.62	2.00	0.32	2.20	3.91	2.34

Alkaline activator

In this study, two different types of activators were applied. It is the activators NaOH and KOH that are responsible for the reaction. NaOH is accessible in the form of powder, whereas KOH is supplied in the form of pellets. When making the two-part geopolymer, the activator was made by dissolving the powder and the pellets in a solution of tap water. The amount of activator (solids) present in a solution is proportional to the concentration of the solution. The activator will be employed at a concentration of 12M, which is the recommended concentration. The alkaline activator solution was produced first, then allowed to sit for 20 minutes before being combined with the fly ash. Therefore, heat is generated during the interaction between fly ash and activator before the mixing process takes place at room temperature, as shown in the diagram.

Aggregate

The coarse aggregate utilized in this study is a normal grade river sand, and the fine aggregate is a normal grade river gravel, according to the manufacturer. The maximum aggregate size that may be utilized is 20 mm.

Samples and curing method

The concretes were 100 mm cubes in volume when they were produced. Table 2 summarizes the mix proportions for the fly ash geopolymer concretes that were produced as part of this study. The samples were dried for 24 hours at 60 degrees Celsius in an oven. After 24 hours of drying in the oven, samples will be removed from the mould and stored at room temperature in sealed plastic bags until testing can be performed on them. The samples for the chloride migration and permeability experiments were produced in cylindrical moulds measuring 100 mm in height by 50 mm in diameter.

Table 2: Mix proportion design for geopolymer concrete

Fly ash proportion (wt%)	Total binder (kg/m ³)	Coarse aggregate (kg/m ³)	Fine aggregate (kg/m ³)	Activator (Molarity)		Liquid to binder ratio
				NaOH	KOH	
100	400	1150	640	12	0	0.40
				0	12	

Testing Method

The sample is subjected to three distinct types of testing. The purpose of the compressive strength test was to determine the mechanical strength of the sample in accordance with British Standard, Testing Concrete-Part 116: Compressive strength of concrete cube was determined using BS1881: Part 116: 1983 (*BS1881: Part 116: 1983 Method for determination of compressive strength of concrete cube*, 1983), functional groups were identified using Fourier Transform Infrared Spectroscopy (FTIR), and microstructures were identified using Scanning Electron Microscopy (SEM). Compressive strengths of the concretes were evaluated after 7, 14, and 28 days of curing, while FTIR and SEM analysis were performed on paste samples of similar mixtures (without aggregates).

RESULTS AND ANALYSIS

Compressive strength

The compressive strength of fly ash geopolymer concretes is plotted in Figure 1 as a function of the mixing technique and activator. The type of alkali activator used, as well as the one-type and two-type geopolymer, impacted the mechanical performance of fly ash geopolymer.

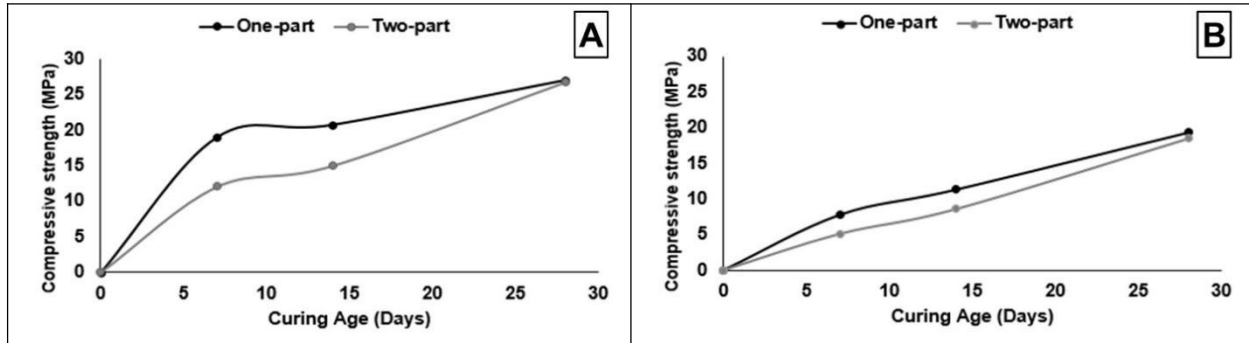


FIGURE 1. Compressive strength development as a function of mixing technique and activator; (A) NaOH, (B) KOH

The compressive strength of fly ash geopolymer concretes using NaOH as an alkali activator is higher than that of fly ash geopolymer concretes with KOH. The type of alkali-activated cement used greatly influences the performance of fly ash geopolymer concrete. In comparison to KOH, NaOH is more often used in the manufacture of geopolymer concrete (Sarker, Kelly and Yao, 2014). The rate of dissolution is one of the reasons why NaOH is more concentrated than KOH (Duxson *et al.*, 2007).

The compressive strength of a fly ash geopolymer including NaOH is enhanced owing to the compact and denser solids, but the compressive strength of a fly ash geopolymer containing KOH is decreased due to increased porosity. NaOH forms a more crystalline structure than KOH (Sarker and McBeath, 2015). Additionally, the production of oligomers in NaOH solution contributes to the increase in compressive strength when compared to KOH, because NaOH has a greater capacity for dissolving alumina silicate minerals (Sarker and McBeath, 2015). The compressive strength of geopolymer concrete will be improved as a result of its well-structured crystalline structure.

One-part fly ash geopolymer concretes showed better strength than two-part fly ash geopolymer concretes. Hajimohammadi, Provis and Van Deventer (2011) show that decreasing the quantity of silica in a one-part fly ash geopolymer enhanced the formation of high-silica crystalline phases. As a result, the initial strength of a one-part fly ash geopolymer is greater than that of a two-part fly ash geopolymer. A geopolymer composed entirely of fly ash was shown to have a higher starting strength but a slower rate of strength growth (Nematollahi, Sanjayan and Shaikh, 2015).

Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectra of the unreacted precursors material utilized in this investigation are shown in Figure 2 with their associated wavenumbers. Wavenumbers between 400 cm^{-1} and 4000 cm^{-1} are detected. It is observed that dehydration had a significant effect on a number of significant alterations in the FTIR spectrum. In alkali-activated paste, the main bands are about 3604 , 1622 , 1072 , 783 , and 462 cm^{-1} .

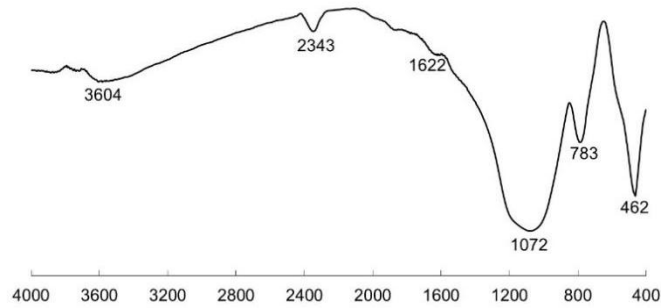


FIGURE 2. FTIR spectra of the unreacted material used to synthesize alkali activated fly ash binders.

Figure 3 illustrates the FTIR analysis of a one-part geopolymers binder, whereas Figure 4 illustrates the study of a two-part geopolymers binder. After geopolymerization, all main bands are reduced except the O-H stretching band. The reactivity of fly ash with various activators is demonstrated by the peak at 980 – 1100cm⁻¹, which corresponds to the development of a binding gel (T-O-T bond) in amorphous glasses. The production of binding gels varies according to the alkali activators used; sodium aluminosilicate hydrate gel (N-A-S-H gel) forms in reaction with NaOH, whereas potassium aluminosilicate hydrate gel (K-A-S-H gel) forms in response with KOH. Table 3 summarizes the band assignments from these FTIR analyses for both one-part and two-part fly ash geopolymers.

The peak number in each band region explained the structural group's bending and stretching. 400–500cm⁻¹ and 750–850cm⁻¹ band areas imply bending vibrations and symmetric stretching of the Si-O-Si bonds, respectively. While the band region between 980 and 1100cm⁻¹ indicates the asymmetric stretching of T-O-T and the development of binding gel. Stretching and bending vibrations of the O-C-O bond at 1410–1420cm⁻¹, stretching and bending vibrations of the O-H band at 1600–3700cm⁻¹ band area with water losses (O-H).

From 7 to 56 days, Figure 3 depicts the micro development of the binding gel in a one-part fly ash geopolymer. The shift in number is significant in the FTIR spectrum because it indicates the dehydration of the materials (Nadziri, Ismail and Hamdan, 2018). Around 3440, 1650, 1420, 1000, and 450cm⁻¹ are the main bands. The wide peak at about 3440cm⁻¹ is attributable to hydrogen-bonded O-H stretching and free water in both types of activator (Figure 3A and 3B) (Nadziri, Ismail and Hamdan, 2018)(Clark *et al.*, 2008). The development of N-A-S-H and K-A-S-H gel is described as a peak in the band region between 800 and 1200. At 7 days, the peak in the pastes with NaOH activator is sharper than those in the KOH activator. It demonstrates that the development of N-A-S-H gel is significantly more than that of K-A-S-H gel after seven days. Then, at 14 days, N-A-S-H and K-A-S-H gels appear to form equally, whereas N-A-S-H gel forms more rapidly than K-A-S-H gel at 28 days. After 56 days of curing, the peak's sharpness on intensity is comparable. Essentially, the steeper the peak, the more N-A-S-H and K-A-S-H gel is produced (Ismail *et al.*, 2013). The wide peaks for K-A-S-H gels are displayed in comparison to N-A-S-H gels. This is due to the porous nature of the materials and their irregular forms (Nadziri, Ismail and Hamdan, 2018), which are also discussed in the next sections (SEM-EDX). Additionally, the wider peaks have a relatively low mechanical strength, as already stated in the preceding section (compressive strength).

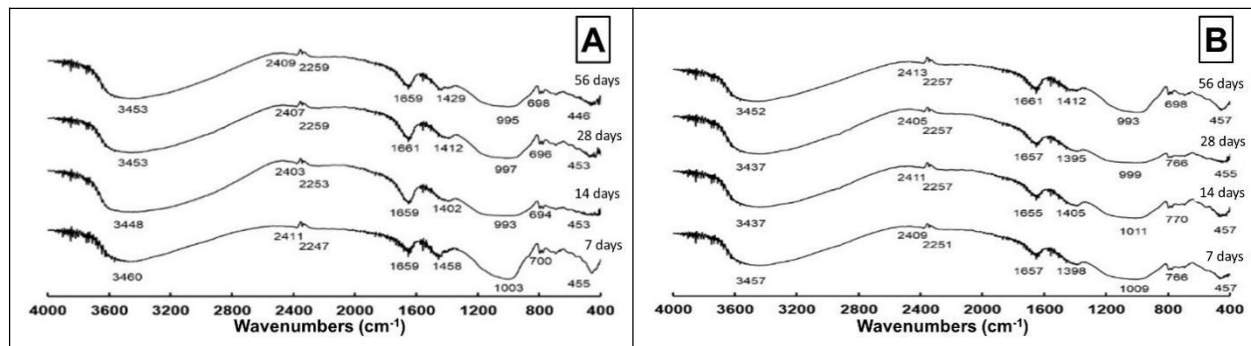


FIGURE 3. FTIR analysis for one-part fly ash geopolymer paste as a function of alkali activator

From 7 to 56 days, Figure 4 illustrates the microevolution of the binding gel in a two-part fly ash geopolymer. Following geopolymerization, all main bands are reduced except the O-H stretching band. The development of N-A-S-H and K-A-S-H gel is indicated by a peak in the band region between 800 and 1200. As the binding gel's microevolution progresses, the peak's sharpness decreases. In general, the higher the peak, the more N-A-S-H and K-A-S-H gel is produced.

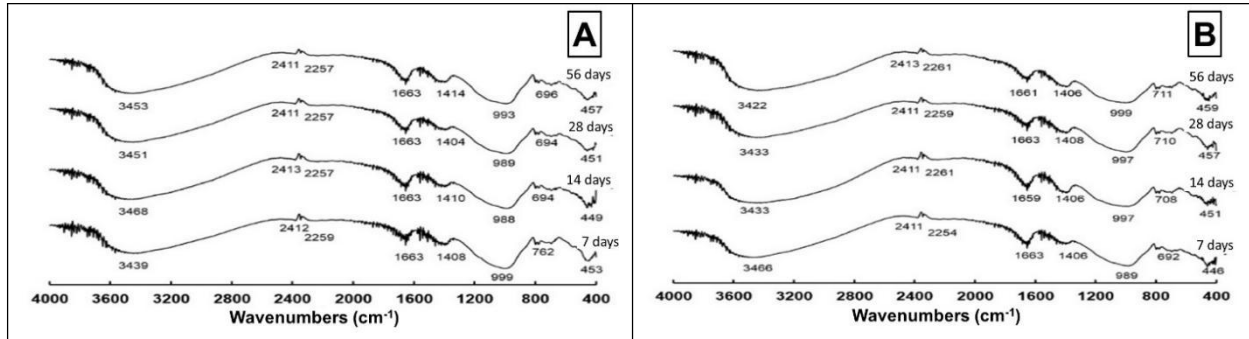


FIGURE 4. FTIR analysis for two-part fly ash geopolymer paste as a function of alkali activator

Table 3: FTIR band assignment for one-part fly ash geopolymer paste as a function of alkali activator

Wavenumbers (cm ⁻¹)	Assignments
400 – 500	Bending vibrations of Si-O-Si and O-Si-O bonds
750 – 850	Symmetric stretching of Si-O-Si
980 – 1100	Asymmetric stretching of Si-O-T (T = Si or Al) (formation of binding gel)
1410 -1420	Stretching of O-C-O
1600 – 1650	Stretching of O-H
2850 – 3700	Bending vibration of H-O-H

Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM EDX)

The microstructure of the raw fly ash utilised in the experiment is shown in Figure 5, which is consisting of spherical particles and predominantly glassy (amorphous) fly ash in nature with very small particles. Reduced friction between aggregate and fly ash particles improves the workability of concrete (Nadziri, Ismail and Hamdan, 2018) & (Pacheco-Torgal, Castro-Gomes and Jalali, 2008). Additionally, the small particle size acts as a filler for voids in geopolymer concrete, resulting in denser and more durable geopolymer materials (Zerfu and Ekapatri, 2016).

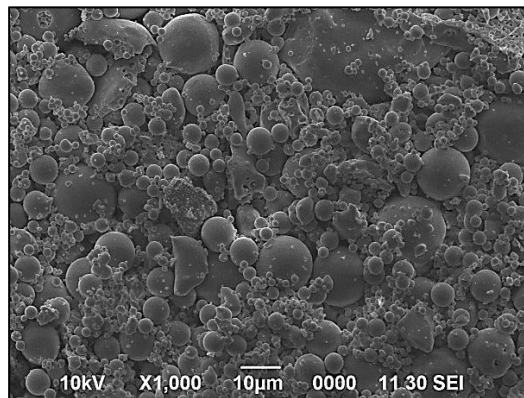


FIGURE 5. Micrograph of raw fly ash

Microstructure of one-part fly ash geopolymer binder

The microstructure of fly ash geopolymer binder was depicted in Figure 6 (A) utilizing NaOH as an activator, whereas KOH. When activated with an alkaline activator, fly ash particles undergo transformation. The changes in the spherical shape of fly ash particles following the alkaline attack can be observed by comparing the morphology of raw fly ash particles in Figure 6. According to Provis and van Deventer (Deventer, 2009), the consolidation of sodium silicate gel with other particles of produced gel during precipitation results in a denser and more compacted cementitious matrix. Additionally, when NaOH is used as the activator, the microstructure is more compact and denser than when KOH is used (Abdul Rahim *et al.*, 2014).

The fly ash particles in Fig. 6 (B) demonstrate that particles are not completely reacted when the spherical shape of unreacted particles is seen. Additionally, there is an empty hollow left by reacting particles, which increases the number of voids and contributes to the material's low mechanical strength. At 14 days, the hollow was filled with reaction product and the amount of reacted fly ash particles increased. While the diameter of the particles grows and they become compacted after 28 days, they do not yet form a dense matrix like the products demonstrated when the NaOH activator is used. This is one of the reasons why geopolymers activated with NaOH has better mechanical strength than geopolymers activated with KOH. As a result of the slower rate of reaction of KOH as an activator, the particles require more time to be completely consumed.

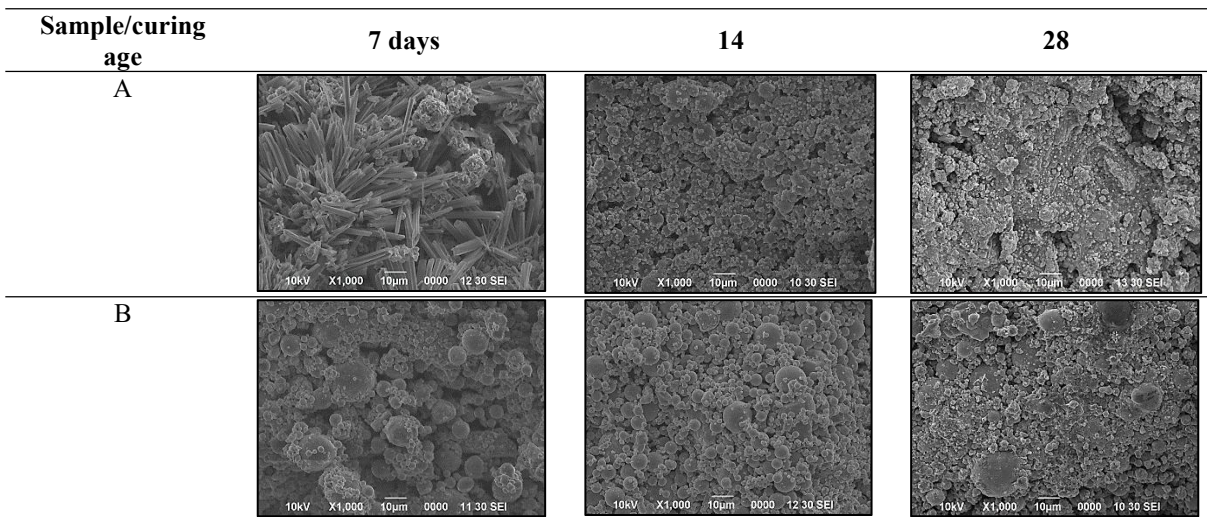


FIGURE 6. Micrograph of one-part fly ash geopolymer paste as a function of curing age and activator: (A)NaOH, and (B) KOH

Microstructure of Two-part Fly Ash Geopolymer Binder

The microstructure of the particles in a two-part fly ash geopolymer binder including NaOH is smaller than the particle size in a one-part fly ash geopolymer binder. At 7 days after curing, SEM images reveal that the particles are not completely reacted, as evidenced by the spherical form of unreacted particles in Figure 8(A). At 14 days, the reaction product began to multiply and create a dense matrix. It therefore, permits the compressive strength to be increased in comparison to 7 days. While at 28 days, the production of compacted matrix was detected. It is defined that fly ash particles are consumed completely after 28 days but require additional time to stabilize the structure due to the denser matrix.

The microstructure of a two-part fly ash geopolymer binder activated with KOH exhibits inconsistent characteristics due to the sample's physical properties during the experiment, which are damp and quickly crack. It then resulted in a lack of strength during compressive strength testing. The visualization of reacted and unreacted particles after seven days of curing is shown in Fig. 8(B). At 14 days, the matrix becomes denser. Even after 28 days of cure, the spherical shape of unreacted particles may be detected under SEM.

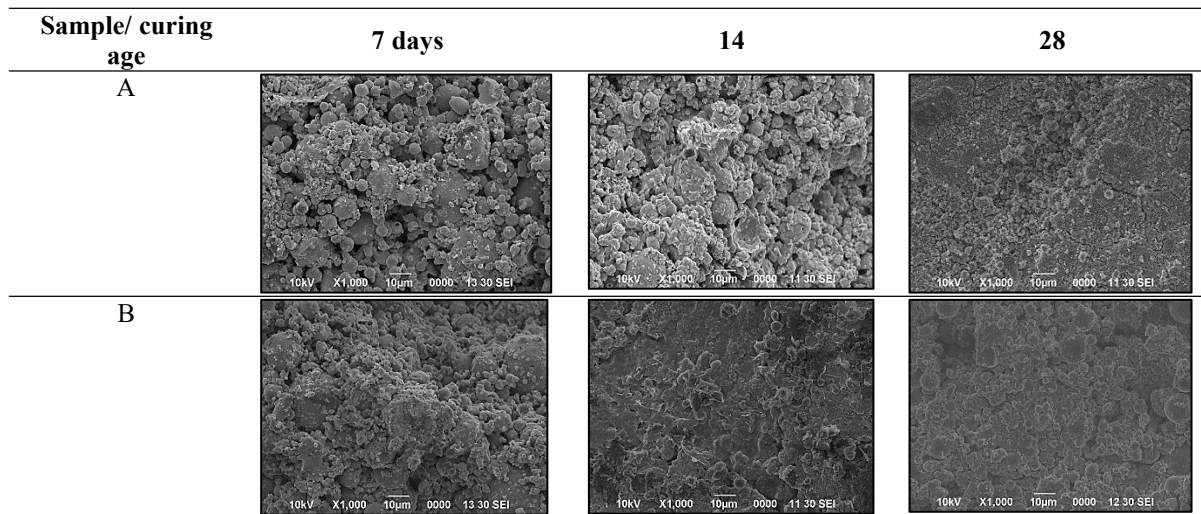


FIGURE 8 Micrograph of two-part fly ash geopolymer paste as a function of curing age and activator: (A)NaOH, and (B) KOH

The compressive strength of fly ash geopolymer concrete was evaluated at the 7th, 14th, and 28th days of concrete age. It can be shown that one-part fly ash geopolymer concrete has a higher compressive strength than two-part fly ash geopolymer concrete. However, the strength of a two-part fly ash geopolymer reached a satisfactory level by the 28th day.

CONCLUSIONS

The compressive strength of one-part fly ash geopolymer concrete is greater than that of two-part fly ash geopolymer concrete. The highest compressive strength and lowest volume of permeable voids are found in one-part fly ash geopolymer concrete. In the concrete mixer, NaOH pellets were combined with fly ash, gravel, and sand. The sodium ions dissolving all silica and alumina solids in the fly ash and the heat released during the hydration of the fly ash and NaOH pellets resulted in high compressive strength and lower porosity.

The fly ash geopolymer binder study reveals that sodium aluminosilicate hydrate (N-A-S-H) gels and potassium aluminosilicate hydrate (K-A-S-H) gels are the binding gels formed in one-part and two-part mixing methods at different alkali activators. Because of the higher presence of Si and Al dissolve when fly ash reacted with the alkali-activator, the N-A-S-H gel and K-A-S-H gel have a higher amount in a one-part compared to two-part fly ash geopolymer binder. The ion dissociates the Si-O-Si and Al-O-Al bonds, allowing Si and Al to enter the liquid phase. Furthermore, the microstructural characteristics of one-part and two-part alkali activators were affected during hydration. The SEM image in one part shows that the particles of NaOH activated fly ash have a more compacted matrix than KOH, and the same is true in the second part. The mechanical strength of the fly ash geopolymer binder will be affected by these microstructural characteristics. As a result, the denser matrix form in particles with higher Si and Al values will have a higher compressive strength.

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