Vol. 14 No. 1 (2023) 19-28

Jurnal Riset
Teknologi Pencegahan Pencemaran Industri

Journal homepage : https://www.jrtppi.id

Kementerian Perindustrian
REPUBLIK INDONESIA

# *Impact of Reduced Activator Concentration and Curing Method on Compressive Strength of Metakaolin/Fly Ash-based Geopolymer Mortar*

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ARTICLE INFO

*Article history:*
Received June 24, 2022
Received in revised form September 14, 2022
Accepted November 22, 2022
Available online May 02, 2023

*Keywords :*
Geopolymer
Fly Ash
Metakaolin
Alkali Activator
Sodium Carbonate

ABSTRACT

The demand for cement is increasing each year, but the manufacture of 1 tonne of cement produces an equal number of carbondioxide ($CO_2$) gas which is directly related to the increase in global warming. Therefore, we need a substitute material, namely geopolymer. This material has relatively superior properties compared to cement. However, one of the drawbacks of geopolymers is that the production costs are relatively more expensive compared to the manufacture of pre-cast cement because it requires chemical solutions such as sodium hydroxide (NaOH) and sodium silicate ($Na_2SiO_3$) to activate the precursor. This research was conducted to replace a specific ratio of alkali activator with water to reduce the use of alkaline hydroxide solutions and sodium silicate while reducing production costs.

The experiment was carried out by replacing the activator solution with water at a certain amount with a different curing method. Mechanical properties, X-Ray Diffraction (XRD), and Fourier Transform Infrared (FTIR) spectroscopy characterization were used to analyze the effect of additional water in geopolymer. The compressive test result shows that the maximum water content that can replace the activator solution is 20% by activator mass for fly ash-based geopolymers and 30% by activator mass for metakaolin-based geopolymers, with sealed and bare curing conditions before the compressive strength was decreased sharply. Substitution of 10% water in fly ash-based geopolymer increases the compressive strength to 17.20 MPa. Compressive test results and characterization showed that the optimal curing condition for fly ash-based geopolymer was sealed curing and bare curing for metakaolin-based geopolymer. The strength increase is due to O-C-O bonds representing sodium carbonate ($Na_2CO_3$), which affects the compressive strength of fly ash-based and metakaolin-based geopolymers.

## 1. INTRODUCTION

One of the primary human needs is a place to live, generally in the form of a house, or other structure, which is used as a place of shelter and residence for a long time. In addition, along with the times, the demand for buildings that support productivity in earning a living, such as offices, factories, warehouses, and other structures, is also increasing. Cement is one of the raw materials in the manufacture of concrete, which in turn becomes the building block of these structures, resulting in the demand for cement production increasing with a ten-fold increase in

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doi : https://10.21771/jrtppi.2023.v14.no.1.p19-28

Accreditation number : (Ristekdikti) 158/E/KPT/2021

global consumption during the last 65 years [1,2]. Cement production is a process that requires much energy because of the high temperature required. Manufacturing 1 tonne of cement requires 4.7 million British Thermal Unit (BTU) of energy, equivalent to burning 400 lbs of coal, which produces about 1 tonne of $CO_2$ [3,4]. Therefore, it is necessary to provide a substitute material for cement that is more environmentally friendly and has a similar compound as cement (pozzolanic materials).

Fly ash is a residual material produced by burning coal. The residual material consists of fine particles carried by flue gas. Fly ash is also the primary component (more than 58%) formed from coal combustion [5,6]. The chemical content of fly ash varies depending on the coal burned, affecting the calcium oxide (CaO) content. Fly ash mainly consists of $SiO_2$, $Al2O_3$, and some trace oxides such as $Fe_2O_3$ and $Na_2O$. Metakaolin is a calcination product of kaolinite clay-based minerals such as kaolin at a temperature of 600°C - 850°C [7,8]. In this kaolin calcination process, the water between the $SiO_2$ and $Al2O_3$ layers will evaporate and change the kaolin into an amorphous phase, namely metakaolin. Both of these materials are common precursors in the formation of geopolymers [9,10]. Geopolymer is a zero cement material consisting of a chain or network of mineral molecules connected by covalent bonds. These chains generally consist of molecular units that contain elements such as silicon (Si), aluminum Al), and oxygen (O). The manufacturing process of geopolymer is called geopolymerization, which is strongly influenced by temperature and time.

The use of fly ash-based geopolymers and metakaolin-based geopolymers as cement substitutes is increasing because they are more environmentally friendly. The physical, chemical, and mechanical properties of geopolymer are almost the same as those of conventional concrete, even relatively better, such as compressive strength, resistance to hot and cold temperatures, and corrosion resistance [11-15]. Geopolymer concrete (GPC) is estimated to reduce 80% of the carbon footprint in construction projects compared with ordinary portland cement [16]. The disadvantage of geopolymers is that they are relatively expensive to process because they require an alkali solution to activate the aluminosilicate materials, which usually consist of alkaline compounds, compared to traditional cement-based concrete only needs water to undergo hydration. The expensive process makes the geopolymer final product cost 10-15% more than conventional concrete, which only needs water to hydrate the cement [17-19]. Several studies have already been conducted to substitute alkali activators with other liquids, such as seawater [20]. This study is conducted to replace a percentage of alkali activators with water, which will reduce the cost of the geopolymer manufacturing process. The curing method in this study was also variated. The effect on mechanical properties and microscopical characterization of geopolymer was analyzed using Compressive Test, XRD, and FTIR.

## 2. METHODS

The class-F fly ash was obtained from Suralaya Coal Fired Power Plant Banten. The kaolin was obtained from Bangka and fired at 850°C for 24 hours to obtain metakaolin. The oxides composition of these materials is shown in Table 1 by using X-Ray Fluorescence (XRF) method.

**Figure 1.** Fly ash (left) and metakaolin (right)

**Table 1.** Chemical composition of fly ash and metakaolin (mass%)

| Oxide | Fly Ash | Metakaolin |
|---|---|---|
| $SiO_2$ | 52.30 | 65.00 |
| $Al_2O_3$ | 26.57 | 33.00 |
| CaO | 6.00 | 0.08 |
| $Fe_2O_3$ | 7.28 | 0.56 |
| $Na_2O$ | 1.41 | 0.06 |
| $SO_3$ | 0.70 | - |

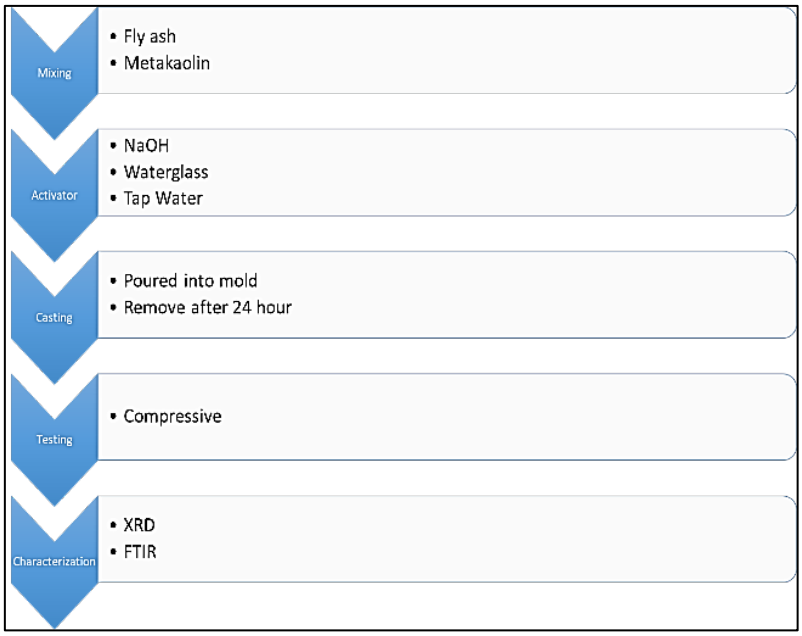


**Figure 2.** Flowchart of Geopolymer manufacturing

**Table 2.** Mix Design of fly ash-based geopolymer

| No | Code | FA (g) | MK (g) | Sand (g) | NaOH (g) | $Na_2SiO_3$ (g) | Water (g) | Curing Method |
|---|---|---|---|---|---|---|---|---|
| 1 | FAS100 | 315 | 0 | 472.5 | 52.5 | 105 | 0 | Sealed |
| 2 | FAS1090 | 315 | 0 | 472.5 | 42 | 84 | 31.5 | Sealed |
| 3 | FAS2080 | 315 | 0 | 472.5 | 31.5 | 63 | 63 | Sealed |
| 4 | FAS3070 | 315 | 0 | 472.5 | 21 | 42 | 94.5 | Sealed |
| 5 | FAS4060 | 315 | 0 | 472.5 | 10.5 | 21 | 126 | Sealed |
| 6 | FAB100 | 315 | 0 | 472.5 | 52.5 | 105 | 0 | Bare |
| 7 | FAB1090 | 315 | 0 | 472.5 | 42 | 84 | 31.5 | Bare |
| 8 | FAB2080 | 315 | 0 | 472.5 | 31.5 | 63 | 63 | Bare |
| 9 | FAB3070 | 315 | 0 | 472.5 | 21 | 42 | 94.5 | Bare |
| 10 | FAB4060 | 315 | 0 | 472.5 | 10.5 | 21 | 126 | Bare |

The sand for light aggregate was obtained from the local building store in Bandung. Technical grades of sodium hydroxide and water glass were purchased from a chemical store in Bandung. The flowchart and mix design for geopolymers is listed in Figure 2, Table 2, and Table 3, respectively.

The method of determining the parameter values in Tables 2 and 3 was based on the main objective of substituting alkali activator solution weight from 0-40%, because higher water content above 40% will disrupt the geopolymerization and specimens will not be hardened. The mass ratio of sodium silicate to sodium hydroxide was kept constant at 2.0. The alkali activator was prepared by mixing a NaOH solution of 12M with $Na_2SiO_3$. Geopolymer was made by mixing fly ash or metakaolin, an alkali activator, and water to create a slurry. It was then poured into a cubical mold with a size of 50 mm x 50 mm x 50 mm, which was in accordance with ASTM C109 "Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)". The specimens were cured under air exposure without protection (bare) and kept in a vacuum bag (sealed) conditions for 28 days. The hardened geopolymer's compressive strength was measured using a tensile testing machine confirmed to ASTM C-39. Debris from compression strength was collected for characterization purposes. The X-Ray Diffraction (XRD)

and Fourier Transform Infrared (FTIR) Spectroscopy measurement was performed at the Instrument and Analytical Laboratory, Chemical Engineering, Institut Teknologi Bandung. The resulting diffraction pattern was compared to the Joint Committee on Powder Diffraction Standards (JCPDS).

**Table 3.** Mix Design of metakaolin-based geopolymer

| No | Code | FA (g) | MK (g) | Sand (g) | NaOH (g) | $Na_2SiO_3$ (g) | Water (g) | Curing Method |
|---|---|---|---|---|---|---|---|---|
| 1 | MKS100 | 0 | 200 | 150 | 113 | 226 | 0 | Sealed |
| 2 | MKS1090 | 0 | 200 | 150 | 102 | 203 | 34 | Sealed |
| 3 | MKS2080 | 0 | 200 | 150 | 90 | 180 | 48 | Sealed |
| 4 | MKS3070 | 0 | 200 | 150 | 79 | 158 | 102 | Sealed |
| 5 | MKS4060 | 0 | 200 | 150 | 68 | 136 | 136 | Sealed |
| 6 | MKB100 | 0 | 200 | 150 | 113 | 226 | 0 | Bare |
| 7 | MKB1090 | 0 | 200 | 150 | 102 | 203 | 34 | Bare |
| 8 | MKB2080 | 0 | 200 | 150 | 90 | 180 | 48 | Bare |
| 9 | MKB3070 | 0 | 200 | 150 | 79 | 158 | 102 | Bare |
| 10 | MKB4060 | 0 | 200 | 150 | 68 | 136 | 136 | Bare |

## 3. RESULT AND DISCUSSION

### *3.1. Compressive Strength*

The compressive test produces data in the form of the maximum load the sample can receive before it is crushed, then the load is divided by the sample's surface area to get the compressive strength of each sample. The compressive strength obtained from the test for each specimen can be summarized in Table 4. In contrast, the breakdown of the effect of each precursor on the compressive strength of the geopolymer is represented in Figure 3 and Figure 4.

The compressive test results of fly ash-based geopolymers are shown in Figure 3. Water substitution for the activator decreases the compressive strength of fly ash-based geopolymer, both in sealed and bare curing. Addition of water will reduce the pH of activator, thereby reducing the presence of the geopolymerization. It also decreases pozzolanic reactivity and produces a non-solid and weak matrix caused by the addition of void spaces and unreacted particles. The compressive strength of fly ash-based geopolymer with bare curing conditions is relatively lower than the sealed cured condition, even with the same amount of water. The low compressive strength is caused by the surface of the geopolymer reacting with free air that has carbondioxide ($CO_2$) gas that reacts with sodium from the geopolymer, producing salt on the surface.

Suppose the curing is carried out for a long time. In that case, the reaction will not only occur on the surface but also propagate below the surface, penetrating the geopolymer, inhibiting the geopolymerization, and weakening the compressive strength.

**Table 4.** Compressive test results

| No | Code | Water content (%) | Activator content (%) | fc (MPa) |
|---|---|---|---|---|
| 1 | FAS100 | 0 | 100 | 34.40 |
| 2 | FAS1090 | 10 | 90 | 27.93 |
| 3 | FAS2080 | 20 | 80 | 16.40 |
| 4 | FAS3070 | 30 | 70 | 1.73 |
| 5 | FAS4060 | 40 | 60 | 0.80 |
| 6 | FAB100 | 0 | 100 | 16.80 |
| 7 | FAB1090 | 10 | 90 | 17.20 |
| 8 | FAB2080 | 20 | 80 | 13.46 |
| 9 | FAB3070 | 30 | 70 | 2.73 |
| 10 | FAB4060 | 40 | 60 | 0.40 |
| 11 | MKS100 | 0 | 100 | 17.60 |
| 12 | MKS1090 | 10 | 90 | 15.60 |
| 13 | MKS2080 | 20 | 80 | 14.70 |
| 14 | MKS3070 | 30 | 70 | 13.86 |
| 15 | MKS4060 | 40 | 60 | 0.46 |
| 16 | MKB100 | 0 | 100 | 22.53 |
| 17 | MKB1090 | 10 | 90 | 18.46 |
| 18 | MKB2080 | 20 | 80 | 15.80 |
| 19 | MKB3070 | 30 | 70 | 12.13 |
| 20 | MKB4060 | 40 | 60 | 0.67 |

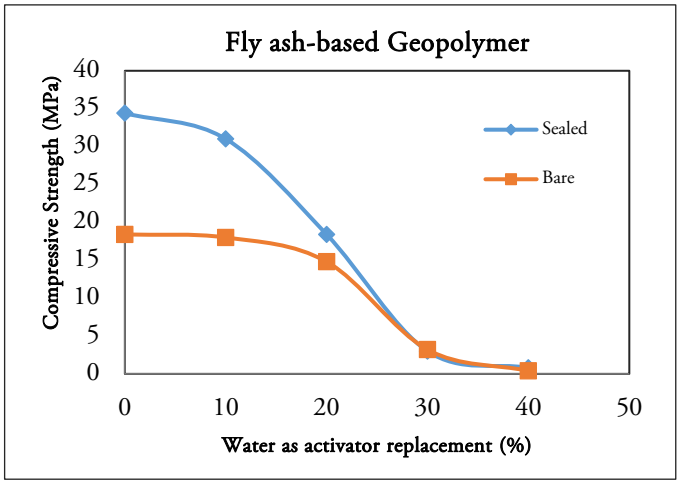


**Figure 3**. Compressive strength of Fly ash based geopolymer

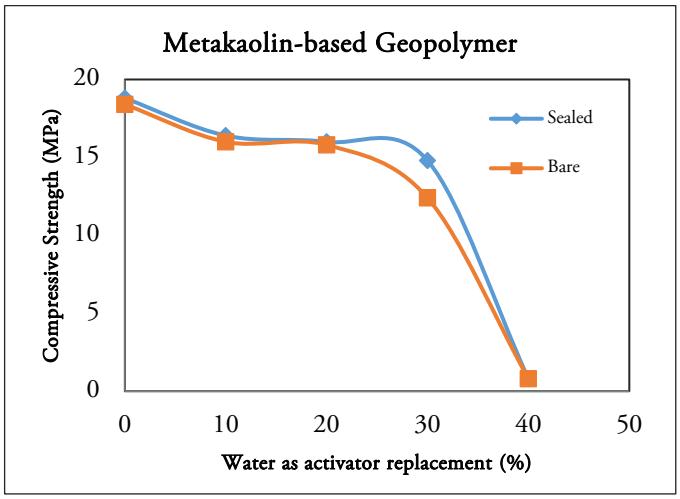


**Figure 4.** Compressive strength of metakaolin-based geopolymer

The compressive test results of metakaolin-based geopolymers are shown in Figure 4. It can be concluded that the compressive strength of the metakaolin-based geopolymer decreased along with an increase in the water content as an activator substitution. The compressive strength reduction is due to the reduced pozzolanic reactivity along with the increase in water content which also happened to fly ash-based Geopolymers in Figure 3 [21]. There is only a slight compressive strength difference between the bare and sealed curing method conditions, which is higher than the compressive strength of fly ash-based in the sealed curing condition. The difference is due to the relatively higher $SiO_2$ content in metakaolin compared to fly ash and relatively less content of other trace oxides so that water does not react with other alkali oxides ($Na_2O$, $K_2O$) to form salts. The less CaO content in metakaolin means the heat generated by geopolymer will be less than fly ash-based. Metakaolin also has a lower density than fly ash, so in the bare curing condition, the water is more easily released through the cavities between the particles, resulting in higher compressive strength. This fact also causes a less significant decrease in compressive strength with the addition of water compared to fly ash-based geopolymers.

### *3.2. XRD Analysis*

XRD characterization was carried out on powders resulting from compression testing with a total water content of 0%, 20%, and 40% for each curing method and type of binder. XRD characterization data that has been processed with XPowder software for fly ash-based geopolymers is shown in Figures 5 and 6.

Figures 5 and 6 show that there are peaks that stand out and differ from the others. These peaks have been compared with the characteristics of the compounds in the XPowder software database. Symbols indicate the compounds formed in fly ash-based geopolymers on the fly ash-based geopolymer above the peak of the curve. The circular symbol (○) indicates the peak characterization of the mineral quartz, the star (☆) symbol indicates the peak characterization of the compound sodium alumino silicate hydrate or $Na_6(Al,Si)_{6.4}H_2O$, and the square symbol (□) represents the mineral albite. Quartz is one of the polymorph of $SiO_2$ contained in fly ash.

XRD results still show the presence of quartz because the SiO2 content in the geopolymer comes from sand aggregate in the most significant amount in a geopolymer relative to the mass ratio. The compound sodium alumino silicate hydrate (NASH) has a chemical formula similar to the chemical formula for geopolymers in general, thereby proving the presence of geopolymer compounds (Na-K)-n(Si-O-)-(Si-O-Al-) or geopolymerization in each batch of fly ash-based geopolymers. The result shows that even though the amount of water is increased, the activator will still be able to react with the binders and fillers sufficiently to form

geopolymers, but in less activation capacity so that they cannot produce geopolymers with high compressive strength. Similarly, albite ($NaAlSi_3O_8$) is a mineral that contains similar elements to a geopolymer structure, which are Na, Al, Si, and O.

XRD characterization data that has been processed with XPowder software for metakaolin-based geopolymers is shown in Figures 7 and 8. Similar to the characterization results on fly ash-based geopolymers, some peaks stand out more than others, and these peaks are identified using X-Powder software as certain compounds. The Quartz, NASH, and Albite are used as indicators for the formation of geopolymers or the occurrence of geopolymerization with two different binders.

Both metakaolin and fly ash are aluminosilicate compounds with almost the same Si:Al ratio (2:1), so the compounds formed after the geopolymerization process are similar. $SiO_2$ compounds were still detected because the amount of Si in the composition of the mixture that formed the metakaolin-based geopolymer is large. It contains metakaolin, sodium silicate, and also from sand. The intensity of $SiO_2$ was increased along with the addition of water content in the activator caused by the non reactant of the remaining $SiO_2$ compounds from metakaolin and sand, thereby reducing the compressive strength of the geopolymer. Figure 5-8 shows that quantitatively, the geopolymer with a pure alkali activator has a more amorphous phase than the geopolymer that uses water as a partial alkali activator replacement. The result explains why both compressive strengths generally decreased because water increased the crystallinity of the resulting geopolymer.

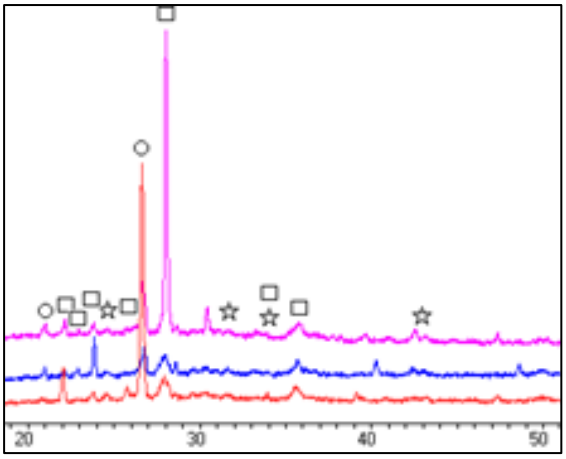


**Figure 5.** XRD diffractogram of fly ash-based geopolymer with sealed curing

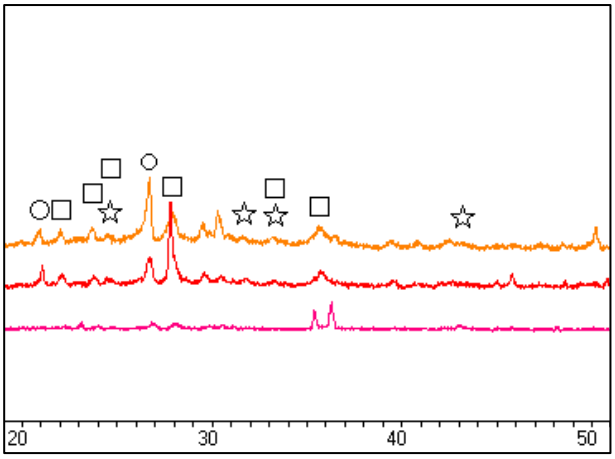


**Figure 6.** XRD diffractogram of fly ash-based geopolymer with bare curing

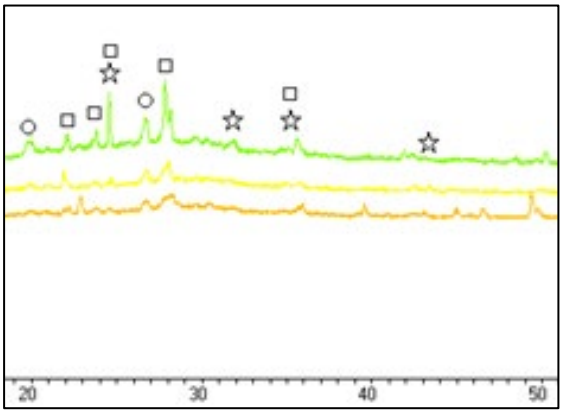


**Figure 7.** XRD diffractogram of metakaolin-based geopolymer with sealed curing

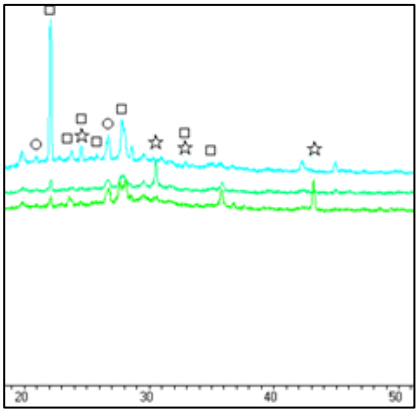


**Figure 8.** XRD diffractogram of metakaolin-based geopolymer with bare curing

*3.3. FTIR Analysis*

The result of FTIR is transmittance data for a wavelength. Each functional group or bond will have a characteristic of absorbing a specific wavelength. The resulting bonds for fly ash and metakaolin-based geopolymers were summarized in Table 5, while the wavelength versus transmittance was presented in Figure 9, Figure 10, Figure 11, and Figure 12. FTIR results have proven that the geopolymerization has successfully occurred due to Si-O-Si and Si-O-Al bonds in the samples.

The stretching Si-O-Si/Si-O-Al on fly ash base material is at wavenumber 1076.08 cm-1, while on fly ash based geopolymer with sealed curing or bare curing conditions, stretching Si-O-Si/Si-O-Al appeared at wavenumber 1012–1024 cm-1. This wavenumber shift indicates the dissolution of Si and Al elements or can be considered as geopolymerization. This result strengthens the previous XRD analysis that even though the water replaces the activator up to 40%, geopolymerization will still occur but not as well as the pure alkali activator and also explains why the compressive strength of the hardened sample is reduced.

The FAS4060 and FAB4060 samples can be seen to have different wavenumber values from other samples for stretching –OH that was caused by the excess water content in the batch, which was 40% of the weight of the activator solution, so it had a –OH bond that looked different from the others, which is the O-C-O bond. The formation occurs due to the reaction of geopolymer with $CO_2$ from free air when the geopolymer is treated by bare curing. The peak formed at FTIR results for the bare cured has a sharper peak at a wavenumber of about 1400 cm-1 than the sealed cured sample. The sharpness of this peak proves that the presence of O-C-O bonds is more significant in the bare cured sample because the sample interacts directly with free air.

Adding water and bare curing conditions will reduce the compressive strength due to excess -OH bonds and O-C-O bonds, both of which have compressive strength below the compressive strength of the Si-OSi and Si-O-Al geopolymer chains. The metakaolin-based geopolymer samples showed peaks in the wavenumber around 1000 cm-1, which indicated the presence of stretching Si-O-Si/Si-O-Al. There are also peaks in the wavenumbers range of 500-700 cm-1, indicating symmetric vibration Si-O-Al. The bending vibration Si-O-Si & O-Si-O appeared at wavenumber 400 cm-1. This result proves that a geopolymerization reaction has occurred, although the quantitative value is unknown.

**Table 5.** Summartized FTIR test results

| No | Code | Stretching OH- ($cm^{-1}$) | Bending H-O-H ($cm^{-1}$) | Stretching O-C-O ($cm^{-1}$) | Stretching Si-O-Si/ Si-O-Al ($cm^{-1}$) | Symmetric Vibration Si-O-Al ($cm^{-1}$) | Bending Vibration Si-O-Si & O-Si-O ($cm^{-1}$) |
|---|---|---|---|---|---|---|---|
| 1 | FAS100 | 3450,65 | 1656,85 | 1413,82 | 1014,56 | 771,53 | 453,27 |
| 2 | FAS2080 | 3450,65 | 1656,85 | 1413,82 | 1012,63 | 771,53 | 449,41 |
| 3 | FAS4060 | 3448,72 | 1647,21 | 1406,11 | 1016,49 | 775,38 | 455,20 |
| 4 | FAB100 | 3450,65 | 1647,21 | 1440,83 | 1016,49 | 775,38 | 447,49 |
| 5 | FAB2080 | 3450,65 | 1656,85 | 1417,68 | 1016,49 | 773,46 | 457,13 |
| 6 | FAB4060 | 3448,72 | 1637,56 | 1417,68 | 1024,20 | 775,38 | 447,49 |
| 7 | MKS100 | 3448,72 | 1656,85 | 1404,18 | 1010,70 | 684,73 | 441,70 |
| 8 | MKS2080 | 3448,72 | 1654,92 | 1406,11 | 1006,84 | 686,66 | 451,34 |
| 9 | MKS4060 | 3450,65 | 1656,85 | 1400,32 | 1020,34 | 688,59 | 443,63 |
| 10 | MKB100 | 3450,65 | 1656,85 | 1415,75 | 1010,70 | 682,80 | 451,34 |
| 11 | MKB2080 | 3448,72 | 1656,85 | 1433,11 | 1010,70 | 688,59 | 443,63 |
| 12 | MKB4060 | 3448,72 | 1656,85 | 1450,47 | 1026,13 | 686,66 | 457,13 |

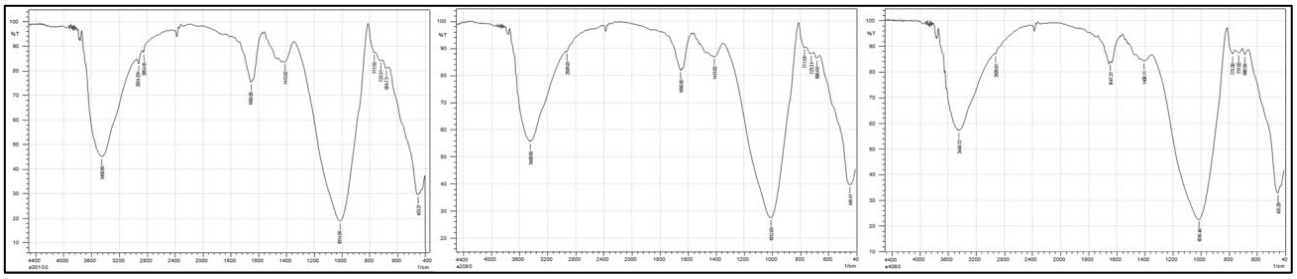

Figure 9. FTIR test results of FAS100, FAS2080, and FAS4060, left to right

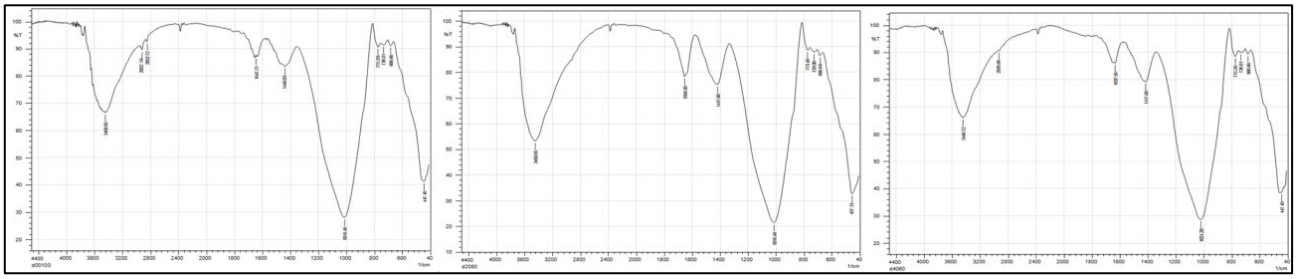

Figure 10. FTIR test results of FAS100, FAB2080, and FAB4060, left to right

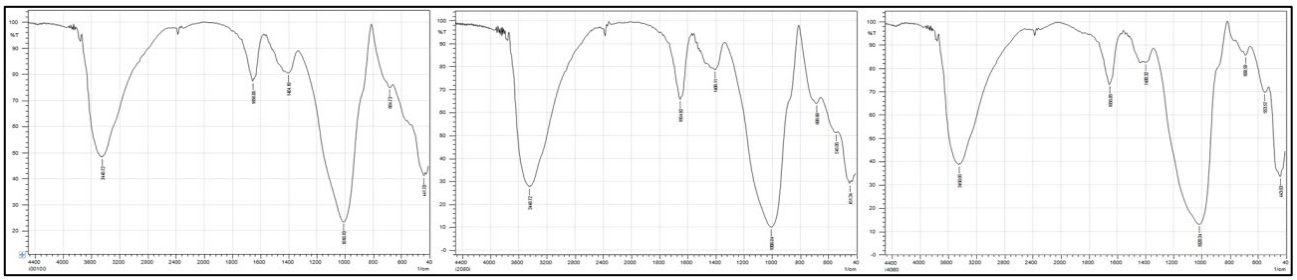

Figure 11. FTIR test results of MKS100, MKS2080, and MKS4060, left to right

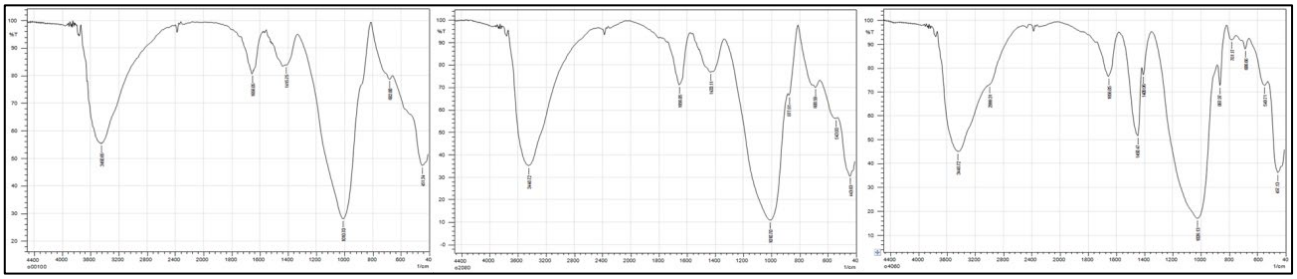

Figure 12. FTIR test results of MKB100, MKB2080, and MKB4060, left to right

The increase in –OH and H-O-H bonds are due to the reaction with water from the free air and the water content in the activator solution in the bare curing and water-containing batch. Trona or sodium bicarbonate is also formed, indicated by stretching O-C-O in each sample. This bond most likely occurs due to the interaction of the sample with $CO_2$ gas contained in the free air. These bonds were detected in all samples at the wavenumber of around 1400 cm-1 on the bare cured metakaolin-based geopolymer sample. It has sharper peaks than the geopolymer cured by the sealed method. The sharper peak indicates a more significant O-C-O bond due to the bare curing condition, which reacts directly with free air.

## 4. CONCLUSION

From this study, it can be concluded that the compressive test results have shown that the maximum water replacement for the activator solution in fly ash-based

geopolymer was 20% under both curing conditions since the addition of higher water content will sharply decrease the compressive strength. However, the activator solution in metakaolin-based geopolymer can be substituted by water up to 30% before undergoing a significant compressive strength decrease. Generally, when water partially replaces the alkali activator, the compressive strength of metakaolin and fly ash-based geopolymer will decrease, except for substituting 10% water in fly ash-based geopolymer, which increases the compressive strength to 17.20 MPa. The difference is due to water having lowered the molarity of NaOH, which is better suited for the fly ash for geopolymerization than a pure alkali activator.

The fly ash-based and metakaolin-based geopolymer compression tests showed that the sealed curing condition resulted in higher compressive strength. The geopolymerization is proven by the XRD characterizations, which showed the formation of NASH and Albite compounds. FTIR characterizations showed the presence of O-C-O bonds as the representation of Trona formed, reducing the compressive strength of Geopolymers.

## ACKNOWLEDGEMENT

We thank Advanced Materials Processing Laboratory, Institut Teknologi Bandung the support for this study.

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