The Analysis of Formation Mechanism of Geopolymer Made From Obsidian Mineral and Its Potential Application as Slow Release Fertilizer

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Abstract

Geopolymer is the inorganic polymer that is made commonly from kaolin, clay, fly ash, or slag. Obsidian mineral is the new candidate material for geopolymer formation. Obsidian is the material that was used as tools in the ancient era, but nowadays this function has been replaced by various metals. Obsidian consists of cristobalite and sodium aluminum silicate. Obsidian was reacted with the disodium metasilicate (Na$_2$SiO$_3$) to form a mineral-based geopolymer. The analysis of formation mechanism through X-Ray Diffraction and Fourier Transform Infrared Spectroscopy shows that the geopolymer formation from obsidian takes place in two stages. The first stage is the formation of a geopolymer from a reaction among cristobalite, disodium metasilicate, and amorphous aluminum silicates, whereas the second stage is the incorporation of crystalline sodium aluminum silicate into the former geopolymer structure. Geopolymer is usually utilized to form brick or concrete for infrastructure purpose, but in this research the geopolymer is proposed to control the release rate of elements from fertilizer. The result of the potassium release test shows that the geopolymer has slow-release property.
Introduction

Geopolymer is the inorganic polymer that is made from minerals or the waste materials that contain silicates as the main component. The reaction of silicates, from those minerals or waste materials, with disodium metasilicate at the high temperature promotes the formation of the inorganic polymer that is widely known as the geopolymer.\textsuperscript{1,2,3} Geopolymer is commonly used as infrastructure materials, such as brick or concrete materials.\textsuperscript{4,5,6,7} Unlike conventional brick or concrete that needs cement as a binder to make it hardened, the geopolymer can be hardened without the addition of Portland cement. Cement industry consumes a high amount of heat energy and generates carbon dioxide (CO\textsubscript{2}), and other harmful gas such as nitrogen dioxide (NO\textsubscript{2}), and sulfur dioxide (SO\textsubscript{2}), therefore this industry is classified as the non-environmental friendly industry.\textsuperscript{8,9} In contrast, the brick or concrete that is made through a geopolymerization reaction does not need the addition of Portland cement as binder. For this reason, the geopolymer brick or concrete is labeled as environmental-friendly brick or concrete.

The common minerals or waste material used to synthesize geopolymer are kaolin,\textsuperscript{4,10,11} clay,\textsuperscript{5,12,13} fly-ash,\textsuperscript{6,7,14} and slag from the blast furnace.\textsuperscript{15,16} These materials contain silicates as the main components but the mineral composition are usually different. In nature, there are other minerals that contain silicates; one of them is obsidian mineral\textsuperscript{17}. The genesis of obsidian is different from kaolin, clay, fly ash, and slag. Therefore, the mineral composition in obsidian is also different from that of kaolin, clay, and fly ash. The synthesis of geopolymer from obsidian offers new information in the field of geopolymer science. Obsidian, which is also known as volcanic glass, is a rock mineral that had been used ever since the ancient era when humans still lived in the
caves. It was the raw material for the people of ancient times to make knives, spear, and other tools or weapons.\textsuperscript{18,19} The archaeological research shows that obsidian was circulated in Indonesia, Malaysia, Philippine, and Papua New Guinea. Although it was distributed in many places in South East Asia, obsidian sources in South East Asia are located in a certain area such as West Java (Indonesia), Pagudpod, Ngarcrilan (The Philippine) and Lou Island (Papua New Guinea).\textsuperscript{20} A similar pattern can also be found in North-East Asia,\textsuperscript{21} and Europe,\textsuperscript{22} and the American Continent.\textsuperscript{23,24,25} After the metal had been found, the function of obsidian to make such tools was significantly reduced. Nowadays, obsidian is only used as a decorative stone. The existence of silicates in obsidian opens the possibility of the geopolymer formation from obsidian. Therefore, the aims of our work in this paper are to synthesize geopolymer by using obsidian and to analyze the formation mechanism of geopolymer formation from obsidian.

Geopolymer is quite common being used in the production of various bricks or concrete. There are no reports on the application of geopolymer for other purposes or other sectors than the civil engineering sector. Since geopolymer is classified as the polymer, there is a possibility that it can be used for other purposes, such as controlling the diffusion rate of certain atoms or molecules. Polymer can be used as the barrier layer to decrease the flow rate of certain molecules that pass the layer.\textsuperscript{26,27} When fertilizer elements, such as potassium ion, are reached by water it will dissolve readily. If there is certain barrier that is put between water and that element, then the element must pass the barrier through the path of release within the barrier. If the size of the path of release in the barrier is at the molecular level, such as a polymer, then the elements must diffuse before being dissolved into the bulk solution. The rate of the molecules from fertilizer can be very much slowed down by passing the molecule through the polymer layer. This principle can be applied to modify ordinary fertilizer to become
slow-release fertilizer. Slow-release fertilizer is a kind of fertilizer in which its nutrient release rate can be significantly reduced.\textsuperscript{28,29} Slow-release fertilizer is needed because the utilization of the ordinary is not efficient. The release rate of nutrients from ordinary fertilizer is always higher than the absorption rate of nutrient elements by plant roots.\textsuperscript{28,29,30} Therefore the remaining nutrient elements, that cannot be absorbed by plant roots, move toward the groundwater zone and become pollutants in the groundwater.\textsuperscript{31}

In this paper, we report the synthesis of geopolymer material made from obsidian and the analysis to reveal the mechanism of geopolymer formation from obsidian. We also report the application of geopolymer that was made from obsidian mineral as the media to control the release rate of elements from fertilizer. Obsidian based geopolymer has been successfully used as the media to control the release rate of potassium from potassium dihydrogen phosphate (KH\textsubscript{2}PO\textsubscript{4}), which is the compound that represents fertilizer. This paper is important because for the first time, obsidian is made as geopolymer and also for the first time geopolymer is used to form slow-release fertilizer material.

**Experimental**

*Synthesis of geopolymer*

The materials that were used to synthesize geopolymer are obsidian rock (West Java, Indonesia), and disodium metasilicate (Na\textsubscript{2}SiO\textsubscript{3}). The as-received obsidian was broken and ground by using a hammer and a jaw crusher, respectively. The crusher that was used is a laboratory scale jaw crusher. The size of the crushed obsidian is around 2 cm (obsidian pebble). The obsidian pebble was cleaned in distilled water and dried at 80
°C for 12 hours. After drying, the obsidian pebble was milled by using tumbling mill equipment. The mill vial is made of steel with an inner diameter of 10 cm and length 20 cm. The mill vial was filled with 13 steel balls with an average size of 1.5 cm. The rotation speed of the vial was set at 80 rounds per minute (rpm) for 6 hours. The milled obsidian powder was screened to give 200-mesh (74 microns) obsidian powder. 20 grams of obsidian powder was mixed with 5M disodium metasilicate solution at the ratio of obsidian and disodium metasilicate of 6, 7, and 8 gram/ml. These ratios were chosen after several tests were done to find the range of composition of pressed samples that can stand in the water without being broken. Half of the mixture was placed in the stainless steel mold with a diameter of 4 cm and a height of 4 cm. That mixture was hand-pressed manually and a 2.5 cm diameter steel stick was punched slowly to make a 2.5 cm diameter hole with height 0.3 cm. 0.5-gram potassium dihydrogen phosphate was placed into the hole and then the rest of the mixture was added into the mold. The powder in the mold was pressed at mechanical pressure 10 kg/cm2 to produce the tablet with a diameter of 1 cm. After being pressed, the tablet was heated in an air atmosphere cubical dryer at 100, 110, 120, 130 °C.

Analysis of the obsidian and the processed samples

X-Ray Diffraction (XRD) equipment (Shimadzu 7000, Japan) was used to analyze the mineral composition of the as-received obsidian and the processed samples. The samples were carefully pulverized, pressed, and were put on the samples holder before the X-Ray radiation was started. The voltage and current of the X-Ray tube were 40 kV and 30 mA, respectively. The samples were irradiated with CuK-alpha radiation at sampling pitch and scanning speed of 0.02 degrees and 2.4 degrees/minutes, respectively. Wavelength Dispersive X-ray Fluorescence (WDXRF) (Rigaku Supermini
200) with fundamental parameters was used to measure the weight percentage of elements in the obsidian. The anode that was used was palladium anode. The voltage and power of the XRF tube were 50 kV and 200 W. Fourier-Transform Infrared Spectroscopy (FTIR) (Shimadzu IR Tracer 100, Japan) was used to obtain the absorbance-wave number profile of the sample. By using the absorbance-wave number profile, the change in every sub-molecular bond can be traced.

**Potassium release test**

The potassium release test was conducted by sinking the sample tablets in the beaker glass that has been filled with 100 ml de-ionized water for 1000 minutes and stirred gently. Every certain period, 1 ml of solution was taken and diluted. After 1 ml of the solution was taken from the beaker glass, 1 ml of water was added to maintain the volume of solution. The diluted solution was analyzed by using Atomic Absorption Spectroscopy (AAS) (Shimadzu type-AA 7000) to measure the amount of potassium within the sample.

**Result and Discussion**

**Analysis of the obsidian**

The XRD pattern of obsidian presented in Figure 1 shows that broaden peak dominates the pattern. It indicates that the amorphous phase is the dominant phase in obsidian rock. Other than the amorphous phase, the peaks corresponding to crystalline phases have also been detected; they are Cristobalite (SiO$_2$, ICDD No. 01-089-3434) and sodium aluminum silicate (Na(AlSi$_3$O$_8$), ICDD No. 01-089-8574). Cristobalite has
been known as the dominant phase in volcanic lava. It is locally present as discontinuous coatings in andesitic lava and as microlaminated coatings and infillings of fissures.\(^{32}\)

The XRD pattern shown in Figure 1 is supported by the result of elemental analysis measured through XRF. Table 1 presents the XRF analysis of obsidian. Obsidian was composed mainly of silicon, oxygen, aluminum, sodium, potassium, and with small amount of calcium and titanium. The composition of obsidian found in West Java Indonesia is relatively similar to the obsidian found in California, the United States of America, in which silicon, aluminum, and oxygen are the main elements.\(^{17}\)

FTIR pattern of obsidian is shown in Figure 2. Obsidian consists of Si-O-Al stretching, Si-O-Si bending, O-H deformation linked to Al\(^{3+}\), Al-Al-O-H vibration, Si-O vibration, Al-O as Si cage (TO4), H-O-H bending of water, and H-O-H stretching bonds that can be detected at wave number 524, 671, 807, 951, 1177, 1307, 1650, and 3250 cm\(^{-1}\) respectively. This data supports the XRD pattern in Figure 1 in which obsidian consists of phases that have element related to these bonds. Cristobalite has Si-O-Si bond, crystalline and amorphous aluminum silicates have AlO4 bond in Si cage and Al-Al-O-H vibration bond, as well as other bonds that attached to that phases.

**The formation of geopolymer**

The obsidian rock from West Java Indonesia consists of aluminum silicate and cristobalite. Sodium aluminum silicate is the common raw material for geopolymerization reaction,\(^{1}\) and Cristobalite is one of the polymorphs of SiO\(_2\) in which the silica tetrahedra are packed in a three-layer structure.\(^{32}\)

Figure 3 shows the XRD pattern of the as-received obsidian and the mixed and heated obsidian and disodium metasilicate that were heated at temperature 100, 110,
120, and 130 °C for two hours. As received obsidian consist of the amorphous phase, cristobalite, and sodium aluminum silicate. In the heated samples, the peaks corresponding to cristobalite and sodium aluminum silicate also can be found, whereas disodium metasilicate, since it is already amorphous, stays with the amorphous phases.

At heating temperature 100 °C, the peaks corresponding to sodium aluminum silicate and the height of broadening peak corresponding to the amorphous phase have increased. The peaks corresponding to sodium aluminum silicate continue to increase with an increase in heating temperature up to 110 °C, but it starts to decrease as heating temperature increases up to 120-130 °C. Meanwhile, the peaks corresponding to cristobalite decrease significantly as the heating temperature continues to increase, and it seems that the peaks corresponding to cristobalite have disappeared from the XRD pattern of the sample heated at 110 °C. The heights of broadening peaks that correspond to the amorphous phase have also decreased as the temperature increase up to 110 °C, but then they decrease as temperature increase up to 130 °C. The XRD pattern at 130 °C shows that the amorphous phase becomes the dominant phase in the system.

The decreasing and disappearance of peaks corresponding to cristobalite and the increasing of the height of broadening peak corresponding to the amorphous phase indicate that cristobalite has converted to an amorphous phase. Cristobalite is the polymorphs of SiO₂ in which the silica tetrahedra are packed in a three-layer structure. The structure of cristobalite is more open than quartz.³² The open structure of cristobalite allows a certain degree of incorporation of other elements into the crystal structure. Cristobalite reacts with the amorphous disodium metasilicate to form geopolymer. Meanwhile, the increasing of peaks corresponding to sodium aluminum silicate is contributed to the decreasing and disappearance of peaks corresponding to cristobalite. After cristobalite has converted to a geopolymer, which is amorphous,
sodium aluminum silicate becomes the only crystalline phase in the system, but finally, at 130 °C most of the sodium aluminum silicate also converted to the amorphous phase. The analysis of XRD pattern of as-received obsidian and samples heated at 100-130 °C reveals that the formation of geopolymer from obsidian takes place in two stages. The first stage is the formation of a former geopolymer from a reaction between obsidian and disodium metasilicate, then it is followed by formation of the later geopolymer through the reaction between sodium aluminum silicate and the former geopolymer.

The formation of the geopolymer affects the binding energy between elements. The change in binding energy can be revealed through the change in its absorbance after being irradiated with infrared rays. Figure 4 shows the absorbance (FTIR) pattern of the as-received obsidian and the samples that were heated at 100, 110, 120, and 130 °C. The heating of the samples brings the change to the absorbance level along the wavenumber. In general, the level of absorbance of heated samples (compared to its baseline) is relatively lower than that of as-received obsidian. It implies that the polarity of every bond is less than that of as-received obsidian. The decrease of polarity is caused by the change that happens to the molecule where the bonds lie. One of these bonds is Si-O-Si bending, which is the bond that attached to cristobalite. Based on Figure 3 and Figure 4, as obsidian is heated at 100-110 °C, the absorbance peak corresponding to Si-O-Si bond decreases, the XRD peaks corresponding to cristobalite decrease and disappear, and the XRD broadening peak corresponding to amorphous phase increase. These facts indicate that cristobalite, as heated, reacts with amorphous sodium silicate and disodium metasilicates to form an amorphous phase, which possibly is a geopolymer.

The significant decrease in absorbance can also be detected from Si-O-Al stretching, OH deformation link to Al$^{3+}$, and Al-O bonding at wave number 524, 807,
and 1307 cm\(^{-1}\), respectively. Si-O-Al stretching and Al-O bondings (cage structure of AlO\(_4\)) are part of the crystalline sodium aluminum silicate structure and amorphous structure of aluminum silicates. The heating of obsidian up to 100 °C, makes the absorbance peaks corresponding to Si-O-Al and Al-O bonds decrease significantly. It indicates that a reaction involving aluminum silicates molecules has happened. On the other hand, Figure 3 shows that in the XRD patterns of the sample heated at 100 °C, the peaks corresponding to cristobalite decrease, whereas the height of broadening peaks corresponding amorphous phase aluminum silicates increase and the peaks corresponding to crystalline sodium aluminum silicate becomes stronger. All of these are the indication of the reaction involving cristobalite, amorphous aluminum silicates, and disodium metasilicates that produce another amorphous phase. The XRD patterns in Figure 3 also show that at 110 °C, most of the entire cristobalite has reacted to produce amorphous phase and the peaks corresponding to sodium aluminum silicates get stronger. On the other hand, the height of absorbance peak of Si-O-Al and Al-O bond at 100 °C are not significantly different from that of 110 °C. All of these facts indicate that crystalline sodium aluminum silicate did not involve in any rapid reaction at 100-110 °C. The strong peak of sodium aluminum silicate in the XRD pattern of the sample heated at 100-110 °C is contributed by the decreasing and the disappearance of the peaks corresponding to cristobalite.

The crystalline sodium aluminum silicate, although it does not involve in any rapid reaction at 100-110 °C, it reacts at 120 °C. The XRD pattern of samples in Figure 3 shows that at 120 °C the peak corresponding to sodium aluminum silicate decrease and the broadening peak corresponding to the amorphous phase increase. The FTIR pattern in Figure 4 supports the XRD analysis in Figure 3; it shows that at temperature 110-120 °C absorbance peaks corresponding to Si-O-Al and Al-O increase
significantly.

The increase of temperature up to 130 °C, as shown in Figure 3, makes the reaction involving crystalline sodium aluminum silicate more rapid than before. Sodium aluminum silicate decreases rapidly and at the same time the amorphous phase increase rapidly. Finally, at 130 °C the amorphous phase becomes the major phase in the system. The FTIR pattern of the samples heated at 120-130 °C shows that the absorbance peaks corresponding to Si-O-Al and Al-O bonds at 120 and 130 °C are not significantly different. It means that the reaction involving crystalline sodium aluminum silicate is the only significant reaction that happened at 120-130 °C.

The role of sodium silicate in the formation of the geopolymer can be analyzed through XRD and FTIR pattern in Figure 5 and Figure 6, respectively. At 130 °C, disodium metasilicate involved in the formation reaction of an amorphous phase through reaction among cristobalite and amorphous aluminum silicates, which is followed by the reaction among disodium metasilicate, amorphous aluminum silicates, and crystalline sodium aluminum silicate to form another amorphous phase. Figure 5 shows that the increasing of disodium metasilicate, i.e. the decreasing of the ratio of obsidian/disodium metasilicate, makes the peaks corresponding to crystalline sodium aluminum silicate decrease significantly and the formation of the amorphous phase becomes more rapid. Meanwhile, Figure 6 shows that the increasing of disodium metasilicate does not give a significant change to the height of absorbance peak corresponding to Si-O-Si and Si-O-Al bonds, but it gives significant change to absorbance peak corresponding to the AlO4 bond. It reveals that AlO4 bond is part of crystalline sodium aluminum silicate structure that loses its polarity after it is incorporated into the structure of the amorphous phase. The amorphous phase that has been formed is called the geopolymer. Thus, the structure of this particular geopolymer
consists of the polymer network of Na, Al, Si, and O atoms. Based on Davidovits\textsuperscript{1} that has used an empirical formula of Poly(sialates), this geopolymer structure, which is constructed mainly by SiO\textsubscript{4} and AlO\textsubscript{4} tetrahedral network, can be written as follows.

\[ \text{Na}_2(\text{SiO}_2z\text{AlO}_2)_{2}\text{H}_2\text{O} \] \hspace{1cm} (1)

The above analysis based on XRD and FTIR pattern reveals that there are two stages of polymerization reaction. The first stage is the reaction among cristobalite, disodium metasilicate, and amorphous aluminum silicate to produce firstly formed amorphous geopolymer. The second stage is the reaction between crystalline sodium ammonium silicate with the firstly formed geopolymer to produce the secondly formed amorphous geopolymer.

\textit{Potassium Release Test}

The release performance of polymerized obsidian is presented in Figure 7. The figure clearly shows that the entire samples have slow-release properties, i.e. potassium was being released slowly from the processed samples. It means that the processed samples have become the geopolymer. The difference in heating temperature affects the release property of the samples. As the sample heating temperature increase, the rate of potassium being released from the sample becomes slower. The heating temperature makes a slight difference at the beginning of release time but the difference becomes larger as the release time increase. The result of the release test supports the XRD and FTIR analysis, in which it has been concluded that the polymerization reaction has taken place at 100-130 °C progressively. Geopolymer was formed in two stages, i.e. the formation of the firstly formed geopolymer at 100 °C from the reaction among cristobalite, disodium metasilicate and amorphous aluminum silicates, and the
formation of secondly formed geopolymer at 130 °C through the incorporation of crystalline sodium aluminum silicate to the firstly formed geopolymer. This mechanism affects the release property of these geopolymers. The release rate of potassium from the geopolymer increases when the system moves towards the second stage of geopolymer formation.

**Conclusion**

Obsidian consists of sodium aluminum silicate (Na(AlSi₃O₈)) and cristobalite (the polymorphs of SiO₂). Geopolymer has been successfully formed through a reaction between obsidian and disodium metasilicate (Na₂SiO₃) at elevated temperatures. The result of XRD and FTIR analysis shows that the polymerization reaction was taking place in two stages. The first stage is the formation of firstly formed geopolymer through the reaction among cristobalite, disodium metasilicate, and amorphous aluminum silicate. The second stage is the incorporation of sodium aluminum metasilicate into the structure of the firstly formed geopolymer to produce the secondly formed geopolymer. Disodium metasilicate plays role in incorporation of cage structure of AlO4 into the firstly formed geopolymer. The release test applied to the geopolymer that previously has been mixed with KH₂PO₄ shows that the rate of potassium being released from the geopolymer is decreased significantly

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![Fig. 1 XRD pattern of obsidian rock from West Java, Indonesia](image)
Fig. 2 FTIR pattern of obsidian rock from West Java, Indonesia

1. Si-O-Al stretching
2. Si-O-Si bending
3. OH deformation linked to Al³⁺
4. AlAlOH vibration
5. Si-O vibrations
6. Al-O as Si cage (TO4)
7. H-O-H bending of water
8. H-O-H stretching of water
Fig. 3 XRD pattern of the as received obsidian and the mixed obsidian/ disodium metasilicate (at ratio 6/1 gram/ml) that were heated at 100, 110, 120 and 130 °C
Fig. 4  FTIR Pattern of the as-received obsidian and the mixing of obsidian/disodium metasilicate (at ratio 6/1 gram/ml) that were heated at 100, 110, 120, 130 °C
Fig. 5  XRD pattern of the mixed and heated (130 °C) obsidian (O)/ disodium metasilicate (W) at ratio 6, 7, 8 gram/ml
Fig. 6  FTIR pattern of the mixed and heated (130 °C) obsidian (O)/ disodium metasilicate (W) at ratio 6, 7, 8 gram/ml
Fig. 7  The profile of potassium release from mixed obsidian/ disodium metasilicate (at ratio 6/1 gram/ml) that were heated at 100, 110, 120, 130 °C

Table 1. Elemental composition of obsidian from West Java, Indonesia

| Element | Si   | Al   | Na   | K    | Ca   | Ti   | Fe   | O     |
|---------|------|------|------|------|------|------|------|-------|
| % Weight| 36.53| 5.32 | 3.01 | 4.18 | 0.80 | 0.13 | 0.97 | 49.06 |