

# PREPARATION OF CONSTRUCTION MATERIALS FROM SIMULATED LUNAR ROCK SAND USING ALKALI SALTS

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\*Corresponding Author, Received: 16 Dec. 2024, Revised: 09 Jan. 2025, Accepted: 15 Jan. 2025

**ABSTRACT:** In this study, we attempted to prepare construction materials from simulated lunar rock sand using fusion agent made up of components obtained from the lunar surface. Currently, space development is active in many countries. When building on the Moon, it is difficult to bring all the construction materials from the Earth, and there is a need to develop construction materials made from lunar resources. In a previous study, geopolymer cement was successfully prepared by alkali fusion of simulated lunar rock sand with sodium hydroxide (NaOH). However, sodium hydroxide is almost nonexistent on the lunar surface and must be transported from outside. Therefore, in this study, we aimed to develop a construction material by applying alkali fusion to simulated lunar sand using a fusion agent consisting of existing elements on the lunar surface. Solidified products with the strength to maintain their form can be produced using sodium hydroxide (NaOH) and potassium hydroxide (KOH), and products using potassium hydroxide are approximately twice as strong as those using sodium hydroxide. Sodium hydrogen sulfide (NaSH), a solidified body with a domed shape and a hollow interior, can be produced when heated in a vacuum atmosphere at 130 °C.

*Keywords: Simulated lunar rock sand, Alkali hydroxide, Alkali sulfide, Geopolymer, Domed-shape formation*

## 1. INTRODUCTION

With the space industry currently booming, space development is rapidly advancing around the world, and the "Artemis Project," which aims to send a manned mission to Mars and is led by NASA and major countries such as Japan, Canada, Australia, and the European Union, plans to construct a lunar base by 2030 to serve as a base for manned activities. However, if the building materials to be used for the lunar base are transported by rocket from the earth, it will cost a huge amount of money (1.2 million dollars/kg) [1]; therefore, it is necessary to develop new building materials using resources that exist on the moon, based on ISRU (In-Situ Resource Utilization) concept of local procurement of useful extraterrestrial materials on extraterrestrial celestial bodies [2, 3]. Therefore, some attempts have been made to produce construction materials from lunar resources [4-6].

The development of building materials from lunar sand, known as regolith, as a resource was focused on. Lunar regolith is a general term for glass fragments, rock-derived fine particles, and fine powders that are abundant on the lunar surface with thicknesses of several centimeters to several meters. There is some research on the preparation of construction materials from lunar sand. For example, alumina cement was manufactured by volatilizing iron, magnesium, and silicon, which are highly volatile components in the regolith, through heating over 1650 °C under a vacuum atmosphere, requiring high temperature [7, 8]. Sulfur concrete was manufactured by connecting regolith with sulfur extracted from lunar rocks, but

sulfur was melted at 119 - 159 °C close to noon temperature of the moon surface (average: 128 °C) [9-13]. Geopolymer concrete was manufactured using silicate polymer to connect the regolith, but silica polymer must be brought from Earth [14].

This study focused on geopolymer concrete. Geopolymer cement is cement produced from mixing sodium silicate, sodium hydroxide, water and some industrial wastes, such as coal fly ash, blast furnace slag and so on, to have higher properties than Ordinary Portland cement for heat resistance, acid resistance and so on [15], and we can prepare geopolymer cement from crushed stone by-product using alkali fusion [16]. In previous studies, geopolymer cement was successfully prepared via alkali fusion of lunar simulated sand (simulant), which mimics the composition of lunar regolith, using sodium hydroxide (NaOH), and the resulting product has heat resistance and radiation shielding capabilities for use on the moon [17, 18]. This way, silica polymer does not have to be transported from the earth. However, sodium is scarce on the lunar surface and must probably be brought in from outside of the lunar surface. Therefore, we will investigate the development of new building materials from lunar sand using fusion agents consisting of potassium (K), ammonia (NH<sub>3</sub>), and sulfur (S) [19], which are known to exist on the lunar surface.

This study aims to develop a building material that can be used on the surface of the Moon by alkali fusion of lunar simulated sand and mixing it with water using a fusion agent with components that can be extracted on the Moon.

## 2. RESEARCH SIGNIFICANCE

This study was based on cost considerations for the future development of the lunar base using the resources on the moon, for local production on the moon, in order to minimize the resources that need to be transported from Earth. For this purpose, in this study, we attempted to convert simulated lunar rock sand into geopolymer cement using fusion agents consisting of elements that are known to exist on the lunar surface to be mixed with water in a vacuum, although observation and strength tests must be performed under earth circumstances.

## 3. EXPERIMENTAL

### 3.1 Sample

In this study, lunar simulated sand (lunar soil simulant FJS-1, CSP Japan, Inc.), which mimics the composition of lunar regolith, was used as an alternative to lunar regolith. The chemical composition of the lunar simulated sand, which was analyzed by X-ray fluorescence analyzer (XRF) (Epsilon1, PANanalytical), was shown in Table 1. The main chemical composition was SiO<sub>2</sub> (42 %), Al<sub>2</sub>O<sub>3</sub> (14 %), Fe<sub>2</sub>O<sub>3</sub> (23 %), CaO (14 %) and others such as TiO<sub>2</sub>, K<sub>2</sub>O, MnO and so on. The morphology of the lunar simulated sand, which was analyzed using scanning electron microscope (SEM) (JSM-6510A, JEOL), was shown in Fig. 1. The majority of the samples are particles around 100 micrometers in size, as shown in the SEM photo.

Table 1 The chemical composition of the lunar simulated sand

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Others
Content (%)	42	14	23	14	2	5

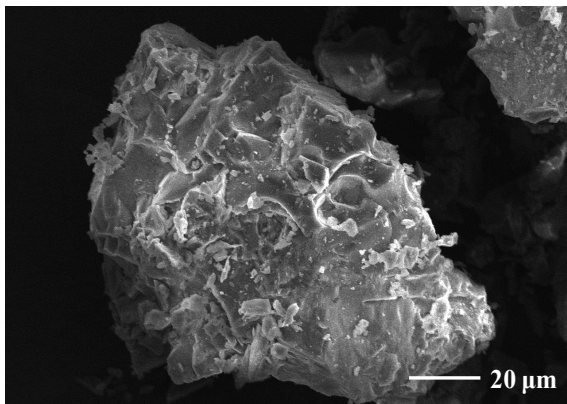


Fig.1 SEM photo of the lunar simulated sand (x 2000)

The fusion agents assumed from the composition obtained on the lunar surface were sodium hydroxide

(NaOH) (Wako), potassium hydroxide (KOH) (Wako), ammonium hydrogen sulfate (NH<sub>4</sub>HSO<sub>4</sub>) (Wako), potassium hydrogen sulfate (KHSO<sub>4</sub>) (Wako), sodium hydrogen monosulfide n hydrate (NaSH·nH<sub>2</sub>O) (Wako) were used. For sodium hydrogen monosulfide n-hydrate, the amount of hydrate was calculated from the thermogravimetric change using a thermogravimetric analyzer (STA-6000, Perkin Elmer) and was approximately 30 % of the total amount.

### 3.2 Preparation of Fused Materials

The experimental conditions were based on previous studies [17, 18]. Experimental procedure for preparation of fused materials from the simulant sand is shown in Fig. 2. Lunar-simulated sand and each fusion agent were mixed at a weight ratio of 1:2 and subjected to alkali fusion treatment in a gas displacement electric furnace (VF-3000) under a vacuum atmosphere at a heating temperature of 200–500 °C for a heating time of 1 h with a temperature rise time of 5 min. Mixing and heating were performed in a platinum crucible, which did not react with the alkali.

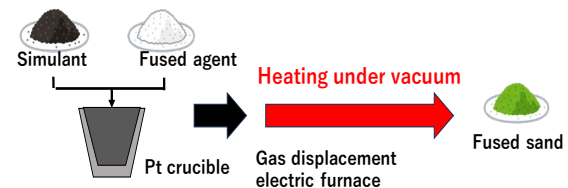


Fig.2 Experimental procedure for preparation of fused material from the simulant sand

The amount of metal dissolved in each fused sand was measured to evaluate alkali fusion using each fusion agent, as shown in Fig. 3. Fused sand (0.1 g) prepared with each fusion agent was added to 1 mol/L HCl solution (20 mL), shaken for 2 h, centrifuged (1200 rpm) for 10 min, and separated into residue and supernatant solutions. The concentrations of silicon (Si), aluminum (Al) and iron (Fe) in the supernatant were measured using an atomic absorption spectrometer (Perkin Elmer, AAnalyst 200), and the dissolved amount of each metal was calculated using Eq. (1).

$$E = C \cdot V / w \quad (1)$$

where  $E$  is the amount of each metal dissolved (mg/g),  $C$  is the measured concentration in the supernatant (mg/L),  $V$  is the solution volume (L), and  $w$  is the weight of fused sand (g).

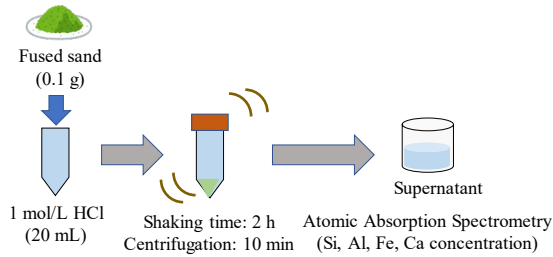


Fig.3 Experimental procedure for the amount of metal dissolved in each fused sand

### 3.3 Preparation of Geopolymer Cement

Experimental procedure for preparation of geopolymer cement is shown in Fig. 4. Among the fusion agents, sodium hydroxide (NaOH) and potassium hydroxide (KOH), which are hydroxide-based, were mixed in a platinum crucible at a weight ratio of 5:1 to 3:1 of lunar-simulated sand and fusion agent, and the fused sands were prepared under vacuum atmosphere using a gas displacement electric furnace at heating temperature of 500 °C with temperature rise time of 5 min and heating time of 10 min. The fused sand and water were mixed in a weight ratio of 5:0.25 to 5:2 to prepare the paste and formed into a mold ( $\Phi 30$  mm  $\times$  5 mm). The geopolymer cement was prepared using a gas displacement electric furnace at a heating temperature of 130 °C, a temperature rise time of 10 min, and a heating time of 3 h under vacuum.

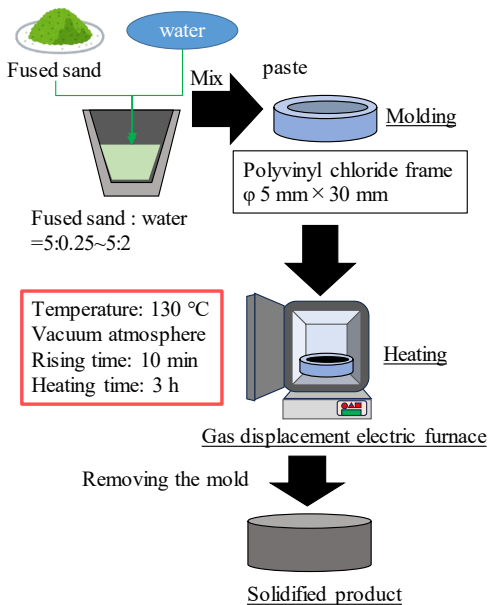


Fig.4 Experimental procedure for the preparation of geopolymer cement

The compressive strengths of the prepared geopolymer cements were tested using a strength tester (ZT series ZTA-500 N, IMADA), as shown in Fig. 4. For the strength test, a pointed attachment

(85°) was attached, and the center of each sample was compressed at a speed of 50 mm/min to measure the strength of failure. For the measurement, the compression test of the strength test method of JIS Z 8841 was used as a reference [20].

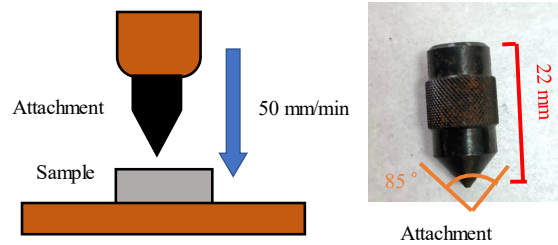


Fig.5 Strength test for the prepared geopolymer cement

### 3.4 Preparation of Sulfur Solidified Object

Experimental procedure for the preparation of sulfur solidified object is shown in Fig. 6. Lunar-simulated sand and a sulfur-based fusion agent, sodium hydrogen monosulfide n-hydrate (NaSH·nH<sub>2</sub>O), were mixed in a platinum crucible in a weight ratio of 1:2. The fused sand was prepared under a vacuum atmosphere using a gas displacement electric furnace at a heating temperature of 300 °C, temperature rise time of 5 min, and heating time of 1 h. The fused sand and water were mixed in a weight ratio of 3:0.5 3:2 to prepare the paste, formed into a mold ( $\Phi 30$  mm  $\times$  5 mm), and solidified using a gas-substituted electric furnace under vacuum and atmospheric atmosphere at heating temperatures of 25, 80, and 130 °C, a temperature rise time of 2 h, and a heating temperature of 1 h.

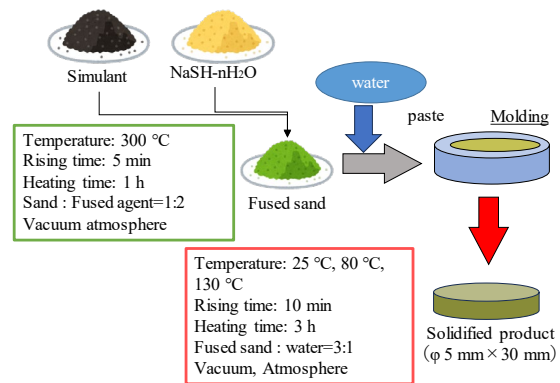


Fig.6 Experimental procedure for the preparation of sulfur solidified object

## 4. RESULTS AND DISCUSSION

Figure 7 shows the dissolved amounts of metal ions of silicon (Si), aluminum (Al) and iron (Fe) in the fused sand generated from lunar-simulated sand using the hydroxide systems, sodium hydroxide

(NaOH) and potassium hydroxide (KOH), the sulfur systems, ammonium hydrogen sulfate ( $\text{NH}_3\text{HSO}_4$ ), potassium hydrogen sulfate ( $\text{KHSO}_4$ ) and sodium hydrogen monosulfide n-hydrate ( $\text{NaSH}\cdot n\text{H}_2\text{O}$ ), as a fusion agent. For sand fused with KOH, the dissolved amount of silicon increased with increasing temperature, and the highest dissolved amount was 103.36 mg/g at 500 °C among all fused sands. For NaOH, the amount of dissolved silicon increased and remained constant above 400 °C, suggesting that a fusion reaction occurred above 400 °C.

Similar dissolution amounts were observed for aluminum, iron, and silicon, suggesting that hydroxide-based fusion agents can produce fused sand with sufficient dissolution at 500 °C. In the sulfur-based fusion agent, the dissolution of silicon and aluminum could not be confirmed, and in iron, NaSH was found to dissolve approximately half as much as KOH.

From these results, sand fused with hydroxides, NaOH and KOH, was used for the preparation of geopolymers to compare the products with NaOH and KOH. In addition, sand fused with NaSH has been used as a sulfur-based fusion agent for the preparation of sulfur-solidified objects.

The paste prepared from sand fused with NaOH and KOH with water addition are illustrated in Fig. 8. For low water-mixing ratio, the fused sand mixed with water was sandy because the formation of alkali-silica solution was insufficient. With increasing the water-mixing ratio, the paste becomes wet and sticky because water sufficiently dissolved the fused sand.

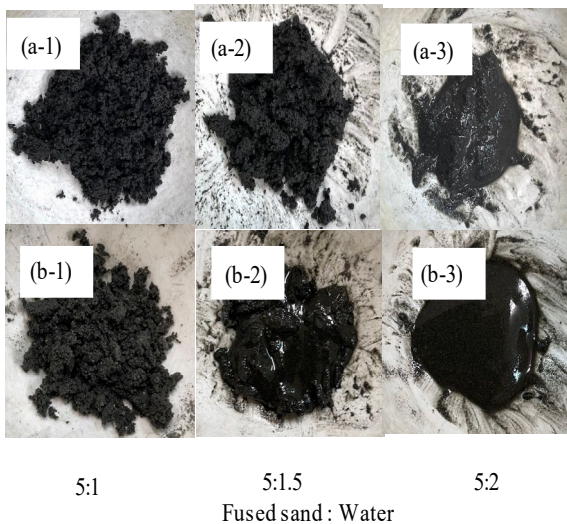


Fig.8 Paste from the sand fused with hydroxide-based fusion agent with water addition: (a) sodium hydroxide, (b) potassium hydroxide

The geopolymer cement prepared from sand fused with NaOH and KOH are illustrated in Fig. 2. Pores appeared on the surface of the geopolymer cement

prepared with NaOH at a water mixing ratio of 5:1 at the points indicated by the red boxes, and the pore diameter and number of pores increased with increasing water mixing ratio.

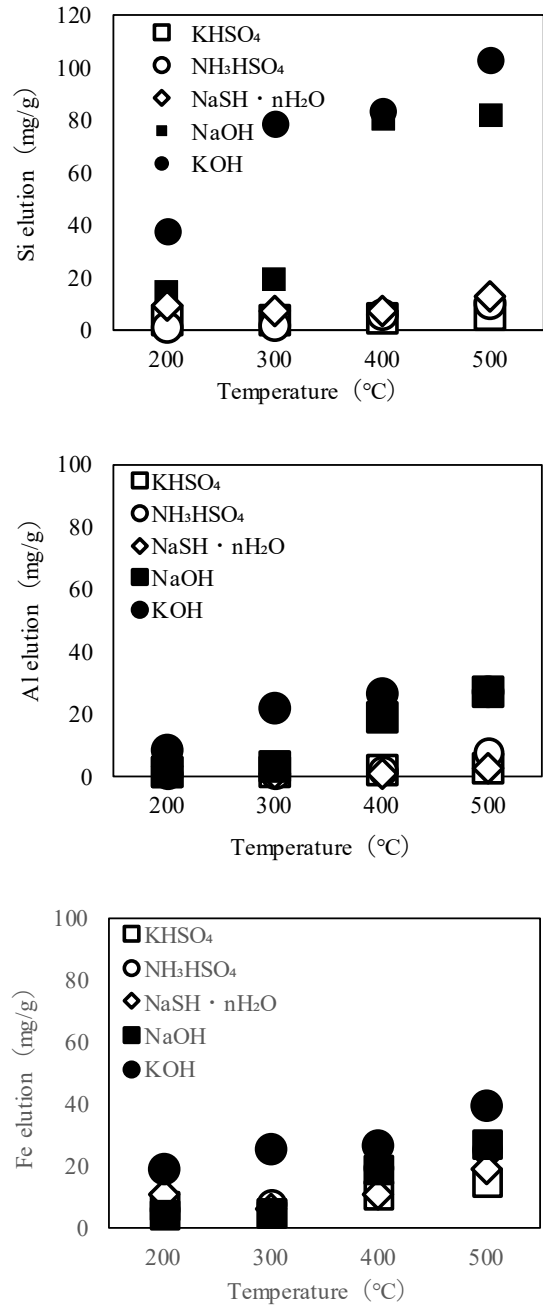


Fig.7 Elution of Si, Al and Fe from the fused sand using each fusion agent at various temperatures

At a water-mixing ratio of 5:2, the material could not maintain its strength during demolding and was crushed. In KOH, pores appeared at a water mixing ratio of 5:1.5, and increased with increasing water mixing ratio; however, the number of pores was significantly smaller than that of NaOH.

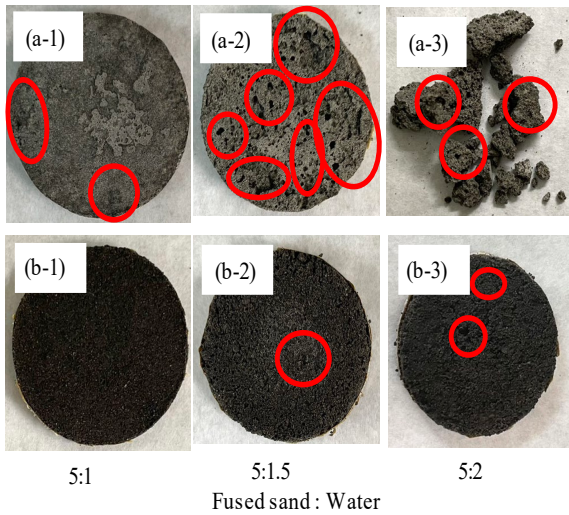


Fig.9 Geopolymer cement from the sand fused with hydroxide-based fusion agent: (a) sodium hydroxide, (b) potassium hydroxide

The strengths of the geopolymer cement prepared from sand fused with sodium hydroxide and potassium hydroxide at each alkali ratio and water mixing ratio are shown in Table 2 and Table 3, respectively. The strength of the cement with NaOH tended to increase with an increase in the water mixing ratio to 5:1. The highest strength (221 N) was obtained at an alkali ratio of 4:1, which was consistent with the results of previous studies. At a water mixing ratio of 5:2, strength measurements were not possible because the geopolymer cement was crushed during the demolding. For potassium hydroxide, the strength of the cement tended to increase at water mixing ratios of 5:0.5 to 1, with the highest strength (543 N) obtained at an alkali ratio of 3:1.

The strength of all geopolymers increased with increasing water content relative to the total, but the strength began to decrease at a water mixing ratio of 5:1.5. This may be due to the fact that the amount of water released to the outside during the dehydration-condensation-polymerization reaction of the geopolymer increased with the increase in water content relative to the total to have some pores in the interior of the hardened geopolymer cement. The overall strength of the geopolymer cement prepared from sand fused with potassium hydroxide was approximately twice as high as that prepared with sodium hydroxide melt. This was because the mixing ratio of lunar-simulated sand and fusion agent was prepared by weight, and the molar concentration of sodium hydroxide in the sample was higher than that of potassium hydroxide because of the difference in molarity.

The cross-section of each geopolymer cement prepared from sand fused with sodium hydroxide and potassium hydroxide and their SEM images are shown in Fig. 10. The geopolymer cement prepared from sand fused with sodium hydroxide had a large

number of pores of various sizes that were widely distributed in the cross section. However, the geopolymer cement prepared from sand fused with potassium hydroxide did not have as many pores as sodium hydroxide, and the interior of the geopolymer cement appeared to be filled.

Table 2 The maximum breaking loads of geopolymer cement from the sand fused with NaOH

Simulant	Fused sand : Water				
	: NaOH	5:0.25	5:0.5	5:1	5:1.5
5:1	33	151	216	93	-
4:1	26	179	221	48	-
3:1	7	177	199	190	-

Table 3 The maximum breaking loads of geopolymer cement from the sand fused with KOH

Simulant	Fused sand : Water				
	: KOH	5:0.25	5:0.5	5:1	5:1.5
5:1	258	470	415	308	83
4:1	134	472	348	301	133
3:1	243	362	543	244	128

The SEM images showed that the cement with sodium hydroxide was irregularly molded with irregularities and pores, whereas that with potassium hydroxide was generally smooth and uniformly molded to be solidified hardly. It has been reported that geopolymers with potassium exhibit increased fluidity and can fill every corner of the mold during fabrication [21]. This suggests that the use of potassium hydroxide as a fusion agent is more effective than sodium hydroxide for compact high-strength geopolymer cement.

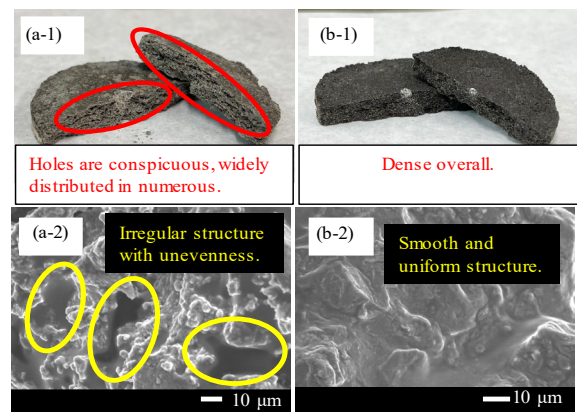


Fig. 10 Cross-section and SEM images of geopolymer cement from the sand fused with hydroxide-based fusion agent: (a) sodium hydroxide, (b) potassium hydroxide

Figure 11 shows sand fused with sodium hydrogen monosulfide n-hydrate (NaSH·nH<sub>2</sub>O) at

various temperatures. At 200 °C and 300 °C, the samples showed an orange material covering the inner side of the crucible with various pore sizes. At 400 and 500 °C, the fused sand was entirely black and adhered to the inside of the crucible. In addition, when the crucible was removed from the electric furnace, the sulfuric odor was stronger above 400 °C than at temperatures below 300 °C because hydrogen sulfide was generated above 350 °C of the boiling point of sodium hydrogen sulfide. When water was mixed with sand fused at 300 and 400 °C, the color and appearance of the sand changed significantly. The sand at 300 °C mostly reacted with the sulfur-based agent, adhering to the inside of the crucible to produce an oil-clay-like paste, while the sand at 400 °C strongly adhered to the inside of the crucible, and almost no paste was collected because the reaction with water could not be confirmed. Therefore, sand fused with a sulfur-based agent at 300 °C was used for the preparation of subsequent sulfur-solidified products.

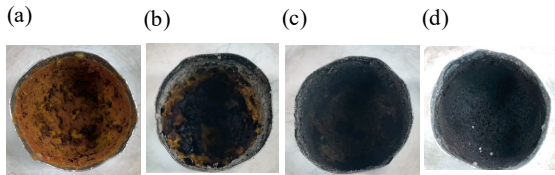


Fig. 11 Sand fused with a sulfate-based fusion agent (a) 200 °C, (b) 300 °C, (c) 400 °C, (d) 500 °C

Solidified specimens were prepared from lunar-simulated sand fused with sodium hydrogen monosulfide n-hydrate at a mixing ratio of 1:2 and heating temperature of 300 °C. The solidified specimens were prepared under vacuum and atmospheric conditions at a melt-to-water mixing ratio of 3:0.5-2, heating temperatures of 25, 80, and 130 °C, a temperature rise time of 10 min, and a heating time of 3 h. Table 4 shows the condition of the solidified specimen after heating under each condition. Under atmospheric conditions, none of the samples solidified or remained in a semi-liquid state (Fig. 12). Under a vacuum atmosphere, the samples treated at 25 °C and 80 °C did not solidify, and only the sample heated at 130 °C solidified. The surfaces of the solidified samples became moist when exposed to air because of the deliquescence of sodium contained in the sample. In some cases, where the amount of water mixed was large and in the sample heated at 130 °C, which is above the melting point of sulfur, the sample overflowed from the top, causing the sample to boil suddenly as the temperature increased rapidly. Therefore, the experiment was conducted again under a vacuum atmosphere with a slower heating rate, rising time of 2 h, heating temperature of 1 h, and heating temperatures of 80 °C and 130 °C.

Table 4 Situation of sulfated solidified specimens under various conditions

Temp. [°C]	Pressure state	Fused sand : Water			
		3:0.5	3:1	3:1.5	3:2
25	Vacuum	X	X	X	X
	Atmosphere	X	X	X	X
80	Vacuum	X	X	X	X
	Atmosphere	X	X	X	X
130	Vacuum	O	O	O	O
	Atmosphere	X	X	X	X

O: Solidified, X: Un-solidified

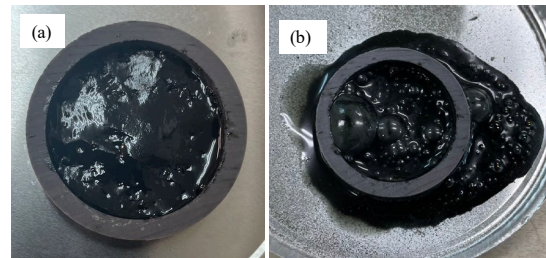


Fig. 12 Specimens with sulfur heating under atmospheric condition: (a) after collection from the furnace, (b) left in room for 3 hours

The appearance of the specimens after heating at 80 and 130 °C is shown in Fig. 13. No signs of overflow from the mold were observed, but the specimens heated at 80 °C were in a semi-liquid state inside and did not solidify. At 130 °C, the specimens produced a dome-shaped solidified body with a hollow interior. This may be due to the fact that the sulfur reached its melting point (112.8 °C) and liquefied.

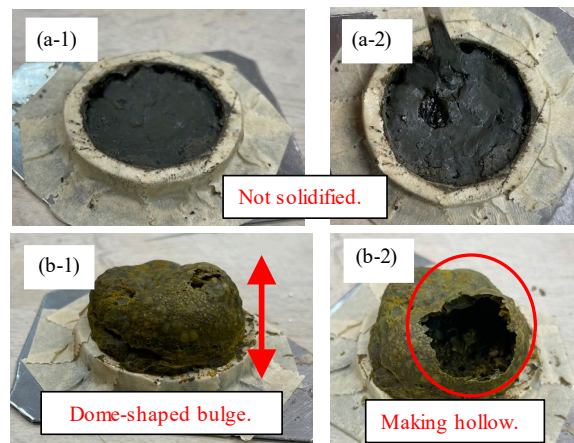


Fig. 13 Specimens with sulfur heated at (a) 80 °C and (b) 130 °C under vacuum atmosphere

Figure 14 shows the XRD patterns of the specimens heated at each temperature under a vacuum atmosphere, and Fig. 15 shows the XRD

patterns of the top and bottom of the dome-shaped sample. It is noted that the original sample peaks, such as quartz ( $\text{SiO}_2$ ), albite ( $\text{NaAlSi}_3\text{O}_8$ ) and anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ ), remain, and those of sulfur minerals, elemental sulfur (S), thenardite ( $\text{Na}_2\text{SO}_4$ ), pyrite ( $\text{FeS}_2$ ), troilite ( $\text{FeS}$ ) and ettringite ( $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$ ), appeared. Sulfur peaks were observed at each temperature; however, at 25 and 80 °C, sulfur was observed in the form of  $\text{S}_8$ , whereas at 130 °C, sulfur in the form of  $\text{S}_6$  was observed. Sulfur has a structure consisting of cyclic  $\text{S}_8$  molecules, the cyclic structure of  $\text{S}_8$  molecules was broken over melting point of sulfur (112.8 °C), and the viscosity of the liquid increases rapidly [22]. The dome-shaped bottom was mostly formed by minerals derived from lunar-simulated sand, and a  $\text{S}_6$  peak derived from the fusion agent was observed in the upper part. This suggests that the sample heated at 130 °C produced  $\text{S}_6$  (9-10 mPa-s) [23], which was more viscous than water (0.2-1.8 mPa-s), and that gases such as water vapor generated inside pushed the sulfur film outward, producing the solidified dome-shaped body. The solidified sample exhibited the same deliquescence.

## 5. CONCLUSIONS

In this study, we attempted to develop a building material that can be used on the lunar surface by preparing fusion sand from lunar-simulated sand, using each fusion agent assumed from the components that are thought to be available on the lunar surface. The solidified products obtained were subjected to compressive strength tests to evaluate their performance as building materials. Geopolymer cement with a maintained strength can be prepared from sand fused with hydroxide-based fusion agents. Its strength was approximately twice as high as that of potassium hydroxide. Potassium hydroxide tended to exhibit higher strength at water mixing ratios of 5:0.5-1. The sand fused with the sulfur-based fusion agent did not solidify under an atmospheric atmosphere, whereas under a vacuum atmosphere, a solidified body with a hollow dome shape inside could be obtained by adjusting the temperature rise time. All solidified specimens under a vacuum atmosphere underwent deliquescence reactions when exposed to air.

From the above, it was confirmed that it is possible to develop building materials from lunar-simulated sand that simulates lunar regolith using a fusion agent that utilizes components that can be extracted from the lunar surface. In the future, a production method with less precious water will be developed by using solvents other than water, and the properties of the obtained geopolymer will be estimated under simulated conditions of the lunar surface, such as heat resistance, low gravity, radiation shielding ability and so on.

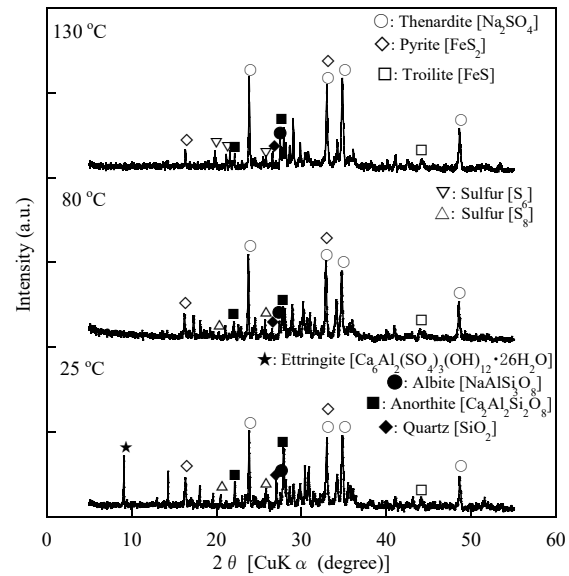


Fig. 14 XRD pattern of sulfate-samples after heating at varying temperatures

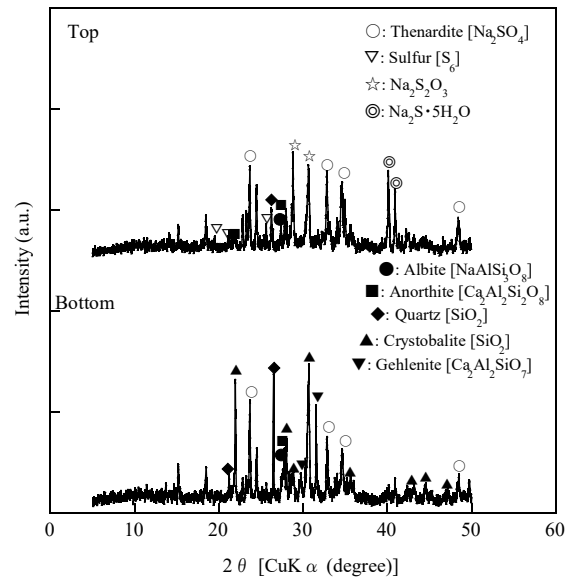


Fig. 15 XRD pattern of the top and bottom of dome-shaped sample

## 6. ACKNOWLEDGMENTS

This work was supported by The Japan Mining Promotive Foundation Survey Test Research Project.

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