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PREPARATION OF GEOPOLYMER CEMENT FROM LUNAR ROCK SAND USING ALKALI FUSION, AND ITS EVALUATION OF RADIATION SHIELDING ABILITY

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ABSTRACT: In this study, simulated lunar rock sand was converted into geopolymer cement by alkali fusion with sodium hydroxide, and its radiation shielding ability was estimated. Space development is currently being conducted in various countries. For construction on the moon, it is difficult to bring all the construction materials from the earth, and construction materials prepared from lunar resources are required. In addition, it is necessary to develop construction materials with radiation shielding ability since the lunar surface is directly affected by space radiation. In a previous study, it has succeeded in making geopolymer cement from simulated lunar rock sand using alkali fusion. Therefore, there is a possibility that geopolymer cement used for construction material can be prepared from lunar rock sand, abundantly present on the lunar surface. In this experiment, geopolymer cement was prepared from simulated lunar rock sand by changing the mixing ratio of the sand, sodium hydroxide and water, and strength of the cement was examined to obtain high-strength geopolymer. In addition, the resulting high-strength geopolymer cement was subjected to radiation shielding tests to confirm its radiation shielding ability. As a result, it was found that the high-strength geopolymer cement with radiation shielding ability can be prepared from simulated lunar rock sand using alkali fusion with sodium hydroxide.

Keywords: Simulant, Alkali fusion, Geopolymer cement, Radiation shielding, Maximum breaking load

1. INTRODUCTION

In 1969, the world's first man landed on the moon, and almost 50 years have passed since then, and the challenge of space continues. Currently space exploration with unmanned spacecraft for space development is active around the world. However, unmanned exploration has its limitations, so manned space exploration is necessary. NASA plans to conduct "the Artemis Program" with the goal of a manned exploration of Mars, which is envisioned as a series of repeated visits to the moon's surface [1]. Therefore, the development of the lunar surface will be very important in space exploration. In space agencies around the world, the moon planned to be developed as a base for space development, because the moon is satellite of the planet earth, the nearest astronomical object. However, if all construction materials for the base construction were to be transported from the earth [2], the cost would be enormous (approximately 100 million yen/kg). Therefore, the development of construction materials made from lunar resources is required.

In this study, we focused on lunar rock sand (regolith) which is abundant on the lunar surface. Regolith is the sand that covers the surface of the moon with a thickness of several centimeters to several meters and is mainly composed of SiO_2 and Al_2O_3 in the form of aluminosilicate minerals, such as anorthite, albite and so on. And, we focused on making geopolymer cement with regolith. On earth, fly ash, mainly contains silica and alumina, is used in making geopolymer [3]. Regolith composition is like fly ash, which is used as a material for geopolymer preparation on earth [4]. In our previous study, the authors have succeeded in making geopolymer cements from lunar simulated sand (simulant) using alkali fusion [5 - 12]. Although ordinary Portland cement is mainly solidified by the formation of needle-like calcium silicate hydrate (C-S-H), geopolymer cement is mainly solidified by the polymerization of silicate ions bridged by metal ions, such as Al^{3+}, Fe^{3+} and so on, which is similar to the reaction of zeolite synthesis [13, 14]. It is possible to make geopolymer cement which can be used as a building material from lunar rock sand abundantly present on the moon by alkali fusion of lunar rock sand to supply silicate ions and metal ions.

On the other hand, space radiation such as galactic cosmic rays [15], ɤ rays, neutrons and solar flares exists in space. Since there is no atmosphere providing protection from radiation on the Moon, each space radiation falls directly on the lunar surface. For this reason, the development of a lunar base or manned space exploration could have a tremendous impact on the astronauts' bodies. Therefore, the development of construction materials with radiation shielding ability is required for the development of a lunar base, because radiation has a tremendous impact on the human body.

2. RESEARCH SIGNIFICANCE

This study was based on cost considerations for the future development of a lunar base, except for resources that can be obtained on the surface of the moon, and therefore resources that need to be transported from Earth have been kept to a minimum. As a purpose, in this study, we attempted to convert simulated lunar rock sand into geopolymer cement by alkali fusion to be mixed with water setting in vacuum, and evaluated its strength and radiation shielding ability.

3. EXPERIMENT

3.1 Sample

Simulated lunar rock sand (Month soil simulation FJS-1, CSP Japan, Inc.) called simulant was used in this study. The chemical composition is mainly composed of 42% of SiO_2, 14% of Al_2O_3, 14% of CaO, 13% of Fe_2O_3 and 3% of MgO.

3.2 Geopolymer Preparation

The precursors of geopolymer are made by mixing the sample with sodium hydroxide powder in a weight ratio of 6: 1 to 3: 1 (17%～33%), put in a platinum crucible, and heated up to setting temperature (500 °C) for 10 min in vacuum atmosphere. Thereafter, geopolymer cement are made by mixing the precursor of geopolymer with water in a weight ratio 6 : 1 to 3 : 1 (17%～33%), forming into a mold (φ5 mm × 20 mm), heating in vacuum atmosphere at 120 °C for 1 to 24 h to obtain the product. Geopolymer cement preparation method is shown in Fig.1.

Afterward, the differences in the mineral phases of each sample were investigated by measuring X-rays diffraction patterns of geopolymer cement making under each conditions using X-ray Diffractometers (D8ADVANCE, Bruker AXS).

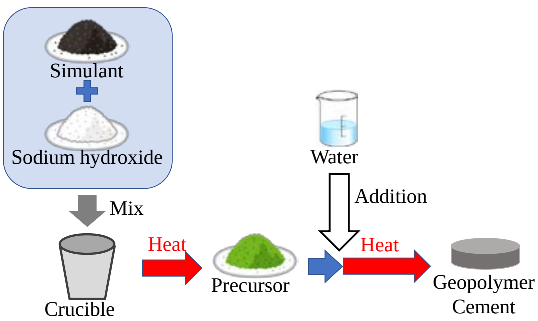


Fig.1 Geopolymer cement preparation method

2.3 Metal Elution Measurement

The precursor of geopolymer (1 g), which was subjected to alkali fusion at each mixing ratio of lunar simulated sand with sodium hydroxide, was put into 1 mol/L hydrochloric acid (20 mL) and shaken for 6 hours, followed by filtration. Metal elution measurement method is shown in Fig.2. The concentrations of silicon, aluminum, iron, and calcium in the filtrate were analyzed by atomic absorption spectrophotometry (Perkin Elmer, AAnalyst 200) and the elution amounts (S) (mg/g) were calculated using the following equation.

$$S = C \times V / w \quad (1)$$

where C is metal concentrations (mg/L), V is filtrate volume (L) and w is the weight for precursor of geopolymer (g).

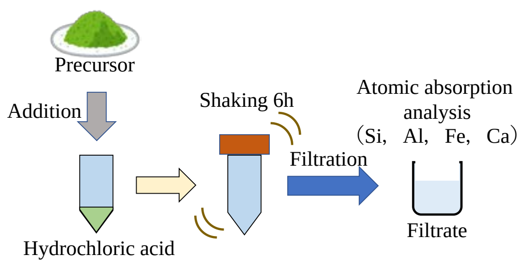


Fig.2 Metal elution measurement method

3.3 Strength Test

The maximum breaking load of the products was measured using a strength tester (ZT series ZTA-500 N, IMADA), referring to the compression test of the strength test method in JIS Z 8841 [16]. Strength test method is shown in Fig.3. The cross section after breaking was observed using SEM (JSM-6510A, JEOL) to analyze the morphology of each sample.

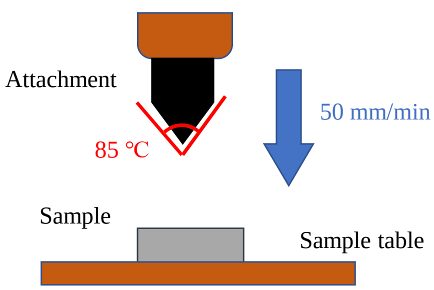


Fig.3 Strength test method

After the strength test, SEM photos for the cross sections of geopolymer cement prepared each condition were taken and compared using Scanning Electron Microscope (SEM, JSM-6510A, JEOL) to investigate the cause of the strength differences.

3.4 Radiation Shielding Ability Evaluation

The product for shielding test (5 cm × 5 cm × 5 mm) was prepared under the conditions to obtain the product with the highest strength, and its radiation shielding ability for γ-rays was evaluated using a radiation measuring instrument (KIND-pro, JSF) and a radiation source (uranium). The experiment was conducted by placing them in a straight line on a flat surface with no unevenness. Ordinary Portland cement (W/C = 50 %, 14 days) was used for comparison of radiation shielding performance. Radiation measurement method is shown in Fig.4. A radiation source (uranium) is placed on a flat surface, and each shielding and measuring instrument is placed on a straight line. By moving the instrument, the distance between the radiation source and the instrument is changed, and the radiation dose at each point is recorded.

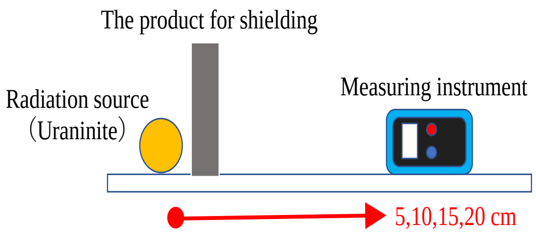


Fig.4 Radiation shielding measurement method

4. RESULTS AND DISCUSSION

4.1 Making Geopolymer Cement

Geopolymer cement was prepared by changing the ratio of sodium hydroxide and water. The XRD patterns of the geopolymer cements prepared with different ratios of sodium hydroxide are shown in Fig.5. The geopolymer cement shown in Fig.5 was prepared with a lunar-simulated sand and sodium hydroxide mixing ratio of 6:1 to 3:1, a heating time of 10 min, and a heating temperature of 500 °C. The melt to water ratio was 20 %, a heating time of 24 h, and a heating temperature of 120 °C.

Gehlenite peaks disappeared in all geopolymer cements samples, and anorthosite peaks decreased with increasing sodium hydroxide compared to the simulant. Although other peak declines are also seen, the behavior of the gehlenite and anorthosite peaks was more significant than the others, suggesting that the reaction of these minerals is a major factor in the geopolymer formation.

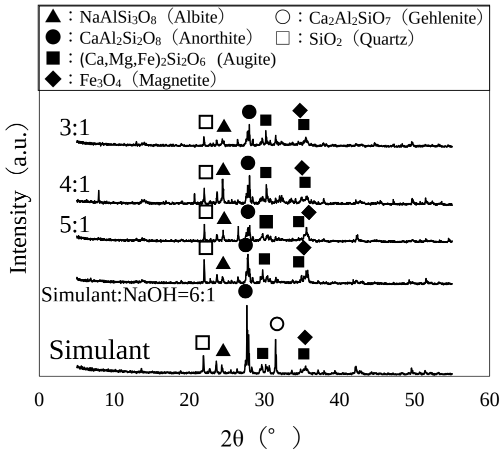


Fig.5 XRD patterns of the geopolymer cements prepared with different ratios of sodium hydroxide

4.2 Maximum Breaking Load

The maximum breaking loads for the products after 24 h heating on various conditions (varying water and alkali ratios) is shown in Table 1. The product with the highest breaking load was obtained when the weight ratio of lunar simulated sand to sodium hydroxide was 4: 1 (25 %) and the weight ratio of the precursor to water was 5: 1 (20 %), indicating 584 N. It is noted that no brittle fracture can be observed on the conditions indicating N.D. As a comparison, the strength of ordinary Portland cement prepared in a size equivalent to the geopolymer cement prepared as a sample for strength test indicates higher than 1000 N. In every making situation, the product of geopolymer cement has a strength approximately one-half or less than that of ordinary Portland cement.

When the water ratio is higher than 25 %, the strength is week (lower than 150 N or N.D.). It is considered that an increase in the amount of water in the mixture causes insufficient hardening by decreasing the bonding of silicates and metal ions, mainly iron and aluminum, due to the dilution, resulting in low strength.

Table1 The maximum breaking loads for the products after 24 h heating on various conditions

	Alkali ratio (NaOH/Simulant)				
Water ratio (Water/Precursor)	(%)	17	20	25	33
	17	421 N	310 N	363 N	319 N
	20	382 N	464 N	584 N	360 N
	25	N.D.	N.D.	N.D.	123 N
	33	N.D.	N.D.	N.D.	N.D.

N.D.: Not detected.

The patterns of strength change of typical geopolymer cements among the prepared samples is shown in Fig.6. The strength of sample with low water content (Fig.6(a)) is higher than that of sample with high water content (Fig.6(b)). Geopolymer cement with low water content showed strong brittle fracture behavior, cracking and fracture centered on the compression point when the highest point was reached (Fig.6(a)). On the other hand, geopolymer cement with high water content exhibited fracture behavior from the start of compaction, with cracks gradually expanding and collapsing around the compression point (Fig.6(b)). As the water content increased, the fracture behavior in strength tests showed no brittleness characteristic of concrete.

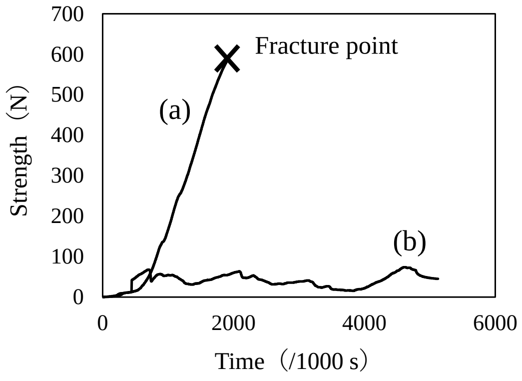


Fig.6 The strength of typical geopolymer cements among the prepared samples; (a) sample with low water content and (b) sample with high water content

The cross section of samples after strength test is shown in Fig.7. Samples with water ratios greater than 25% showed some large holes in the cross section (Fig.7(a)), while those with water ratios less than 25% were almost filling overall (Fig.7(b)). The lower strength of sample with high water is related to the reduction in internal density due to some large holes.

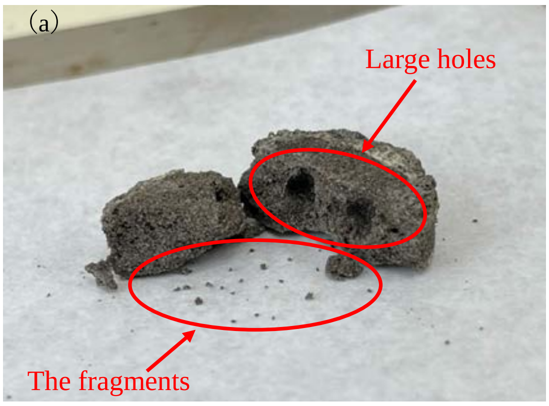


Fig.7 The cross section of samples after strength test; (a) water ratios greater than 25% and (b) water ratios less than 25%

Elution of Si, Al, Fe and Ca from precursor of geopolymer with various NaOH addition is shown in Fig.8. The amount of silicon elution increased as the addition of sodium hydroxide increased. The elution of aluminum and iron were nearly constant, while calcium was almost completely undissolved regardless of the addition of sodium hydroxide.

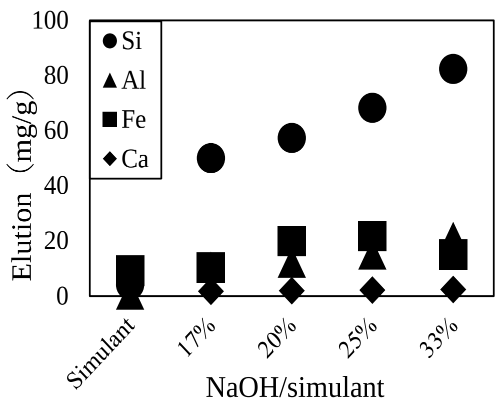


Fig.8 Elution of Si, Al, Fe and Ca from precursor of geopolymer with various NaOH addition

The ratio of the amount of metal dissolved from the precursor of geopolymer with various NaOH addition and the strength of their prepared products are shown in Fig.9. It is noted that calcium is not included in the metal ratio because very little calcium is dissolved from the precursor. The dissolution of aluminum and iron relative to the dissolution of silicon (Si/(Al+Fe)) is smaller, the strength is higher. This is because in the solidification of geopolymer cement, trivalent cations, such as iron and aluminum ions, react with silicate monomers to cross-link each other to form polymers, and when the ratio is smaller, more cross-linking promotes to form polymers to be harden.

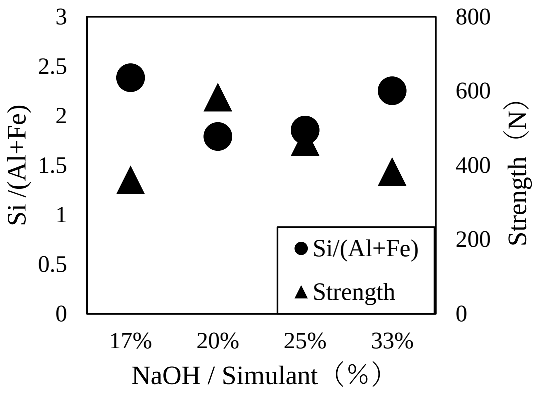


Fig.9 Ratio of the amount of metals dissolved from the precursor of geopolymer with various NaOH addition and the strength of their prepared products

The solidification of the samples was examined by heating the mixture of lunar-simulated sand and sodium hydroxide at a weight ratio of 4: 1 (25 %) and the precursor and water at a weight ratio of 5: 1 (20 %) under a vacuum atmosphere for 1 h to 3 h at 120 ℃. It is noted that 120 ℃ is the average of surface temperature on the moon during the sunshine. The maximum breaking load of the prepared geopolymer cement as a function of heating time is shown in Fig.10. The strength is small from 0 to 2 h and increases to becomes constant (about 200 N) over 2 h.

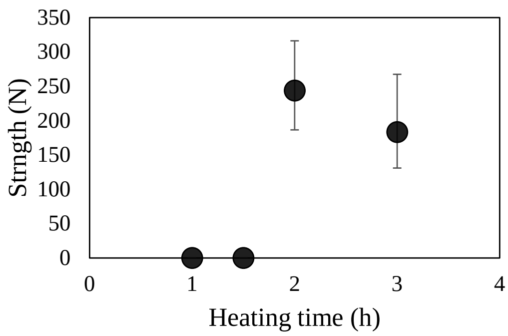
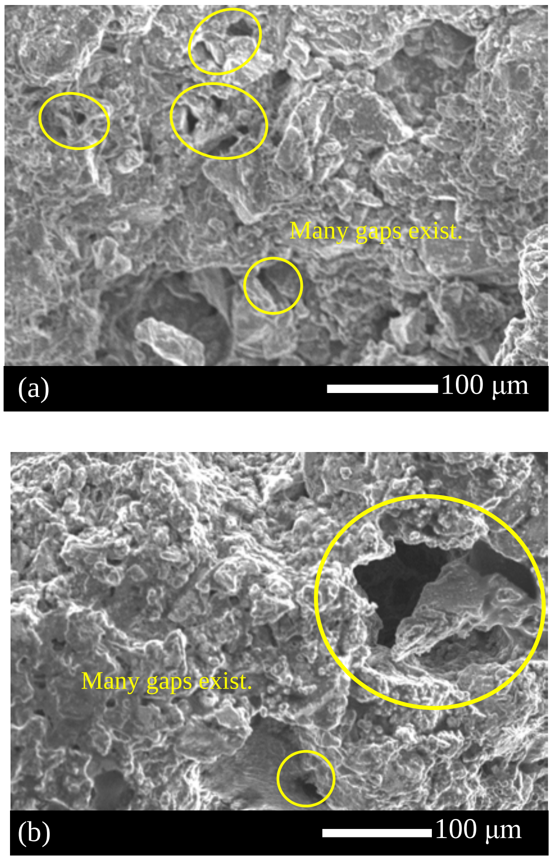


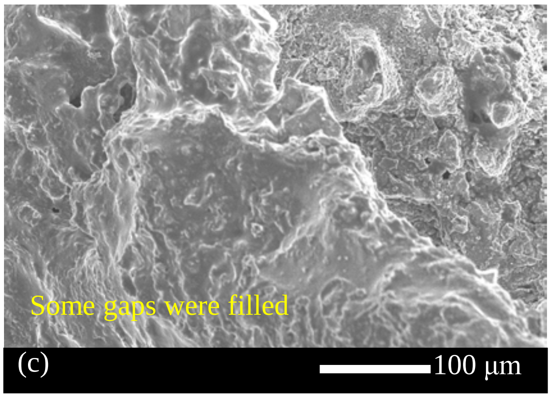
Fig.10 The maximum breaking load of the prepared geopolymer cement as a function of heating time

The photo after heating around 2 hours is shown in Fig.11. wet areas were observed on the surface of the sample after heating at lower than 2h, and the wooden needle pick was stuck on the surface of the sample, while the wooden needles was not stuck on the samples after heating over 2 h. It is considered that insufficient solidification occurred when the heating time was less than 2h due to insufficient dehydration reaction during the formation of the geopolymer, and solidified sufficiently after 2 h heating.

Fig.11 Photo after heating around 2 hours

SEM photos for cross sections of the product prepared by heating at 1 - 3 h are shown in Fig.12. At 1 h and 1.5 h, various sizes of gaps were observed in various places, while at 2 h and 3 h, no large gaps were observed, and the gaps were generally filled. It is considered that gaps were filled by the geopolymer due to the dehydration of water by the effect of longer heating time. Therefore, with the size of the sample of geopolymer cement produced in this experiment, it is considered that at least 2 hours of dehydration would be required. Furthermore, some degree of dehydration reaction may lead to an increase in the strength of geopolymer cement.





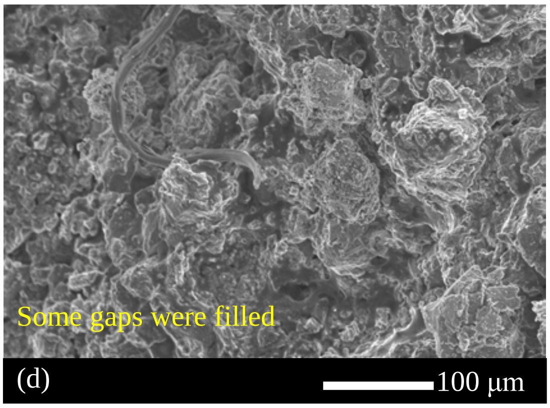


Fig.12 SEM photos for cross sections of products prepared by heating at (a)1 h, (b)1.5 h, (c)2 h, and (d)3 h

4.3 Radiation Shielding Ability

The radiation doses of the prepared geopolymer cement (GP) product, ordinary portland cement (PC), no shielding, and background (BG) are shown in Fig.13. Geopolymer cement has radiation shielding ability since it is lower than no shielding. The radiation shielding capacities of geopolymer cement product were almost same as those of portland cement.

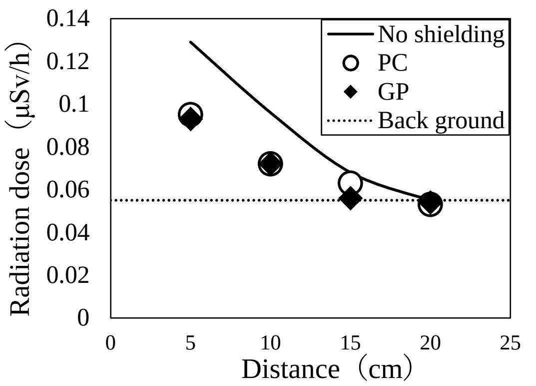


Fig.13 Radiation dose of the product and ordinary portland cement.

5. CONCLUSION

An attempt was made to prepare geopolymer cement by alkali fusion of simulated lunar rock sand with sodium hydroxide. The highest strength was found when the ratio of lunar simulated sand to sodium hydroxide and water was 4 : 1 : 1 (the mixture of lunar-simulated sand and sodium hydroxide at a weight ratio of 4: 1 (25 %) and the precursor and water at a weight ratio of 5: 1 (20 %)), and 2 h heating time in vacuum at 120 ℃ was found to be sufficient for the dehydration reaction in the formation of geopolymer.

The obtained geopolymer cement has the radiation shielding capacity and its capacity was found to be comparable to that of commercially available ordinary portland cement.

6. ACKNOWLEDGMENTS

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