International Journal of GEOMATE, June, 2020, Vol.18, Issue 70, pp. 62 - 67
ISSN: 2186-2982 (P), 2186-2990 (O), Japan, DOI: https://doi.org/10.21660/2020.70.9162
Geotechnique, Construction Materials and Environment

PREPARATION OF GEOPOLYMER CEMENT FROM SIMULATED LUNAR ROCK SAND USING ALKALI FUSION

*Kazuki Sakamoto[1], Takaaki Wajima[1]

[1] Graduate School of Science and Engineering, Chiba University, Japan

*Corresponding Author, Received: 12 Oct. 2019, Revised: 20 Dec. 2019, Accepted: 15 Jan. 2020

ABSTRACT: In this study, it was tried to convert simulated lunar rock sand into geopolymer cement by alkali fusion with sodium hydroxide. 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 development of construction materials made from lunar resources is required. The authors have succeeded in making geopolymer cement from crushed stone dust using alkali fusion. Therefore, there is a possibility that geopolymer cement used for construction material can be made from lunar rock sand, abundantly present on the lunar surface, using the same method. In this experiment, simulated lunar rock sand was fused with NaOH by changing the mixing ratio of the sand and sodium hydroxide, heating temperature, heating time in vacuum or air atmosphere, and the reaction in vacuum and air was compared and examined. As a result, it was found that the elution contents of Si and Al in the fused sand into acid solution increased with increasing the temperature, NaOH addition and heating time of alkali fusion, and the fused reaction in vacuum atmosphere is different from that in air atmosphere.

Keywords: Lunar Rock Sand, Alkali Fusion, Geopolymer Cement, Vacuum Atmosphere

1. INTRODUCTION

Space exploration with unmanned spacecraft for space development is currently active in various countries. The moon is satellite of the planet earth, the nearest astronomical object, and development as a base for space development is planned. In the development plan, when a large-scale base is built on the moon, it is difficult to transport all building materials from the earth [1]. Therefore, the development of building 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 meters and is mainly composed of SiO_2 and Al_2O_3 in the form of alumino- silicate minerals, such as anorthite and augite. In our previous studies, the authors have succeeded in making geopolymer cements from rock dust, discharged from a quarry, using alkali fusion [2 - 8]. 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 [9, 10]. It would be possible to make geopolymer cement which can be used as a building material by using the same method for lunar rock sand abundantly present on the moon.

Therefore, in this study, we tried to make geopolymer cement by reacting simulated lunar rock sand with sodium hydroxide in air and vacuum. We prepared the precursor of geopolymer fused by changing the mixing ratio of sodium hydroxide, heating time and temperature in air and vacuum, and compare the reaction to make geopolymer cement.

2. EXPERIMENT

2.1 Sample

Simulated lunar rock sand (FJS-1, CSP Japan, Inc.) was used in this study. The chemical composition is mainly composed of 42% of SiO_2, 13% of Al_2O_3, 12% of CaO, 15% of FeO and 8% of MgO.

2.2 Preparation of Fused Dust

The sand and sodium hydroxide powder are mixed in a weight ratio of 1: 0.25 to 1: 2, put in a platinum crucible, and heated up to setting temperature (300 to 500 °C) in 10 min in air and vacuum atmosphere. After heating for 0 to 120 min, it cooled naturally at room temperature, recovered fused dust as a powder, and the mineral phase was identified by a powder X-ray diffractometer (MiniFlex 600, Rigaku).

In order to confirm the possibility of making geopolymer cement, the soluble elements, Si, Al, Fe, Mg and Ca, in the sample were examined. 0.1 g of the fused dust prepared under each condition is added into 20 mL of 1 M HCl solution, shaken at room temperature for 6 h, centrifuged, and the contents of Si, Al, Fe, Mg and Ca in the supernatant were measured by an atomic absorption spectrophotometer (Perkin Elmer, AAnalyst 200) to calculate the solubility (S) using the following equation;

$$S = C \times V / w \tag{1}$$

where C is the concentration of each element in the supernatant (mg/L), V is the volume of HCl solution, and w is the weight of the fused sand.

2.3 Preparation of Geopolymer Cement

Preparation of geopolymer cement was performed using the fused sand. The raw sand, water and the fused sand were mixed to prepare the mixtures of 5 g weight, and the mixing ratios used in this study are 1:1:2, 1:1:3 and 2:1:2. The mixture was formed into a mold (ϕ 25 mm $\times$5 mm) and allowed to stand at 80 °C in air atmosphere and cure for 1 or 7 days at 80 ºC. Furthermore, considering the lunar environment, geopolymer cement was prepared under atmosphere of air and vacuum at heating temperature of 80 ºC and 106 ºC (Average monthly temperature).

2.4 Strength Test

The obtained geopolymer cement were subjected to strength tests using a strength tester (ZT series ZTA-500N, IMADA). The sharp point attachment was inserted into the sample at a speed of 30 mm/min to measure the strength when the sample broke. Strength tests for the Portland cement were carried out to compare strength. Portland cement was prepared at water-cement ratio (W/C) = 40 %, formed in a mold and was cured at room temperature for 7 days. Considering the temperature change on the lunar surface, the geopolymer cement cured by heating at 106 ° C for 7 days in a vacuum atmosphere was heated to 200 °C (highest monthly temperature) in an electric furnace or cooled to -196 °C (lowest monthly temperature) in liquid nitrogen for 20 minutes. After heating or cooling, the strength of the geopolymer cement was measured.

3. RESULTS AND DISCUSSION

3.1 Preparation of Fused Dust

3.1.1 Mixing Ratio

The XRD patterns of the fused sands prepared by changing the mixing ratio in (a) air and (b) vacuum are shown in Fig. 1. The experimental conditions are a heating temperature of 500 ° C and a heating time of 60 minutes. In simulated lunar rock sand, the peaks of anorthite and augite were confirmed. In the sands after alkali fusion, the peaks of anorthite and augite in the sand disappeared and the peaks of sodium silicate were confirmed in both air and vacuum.

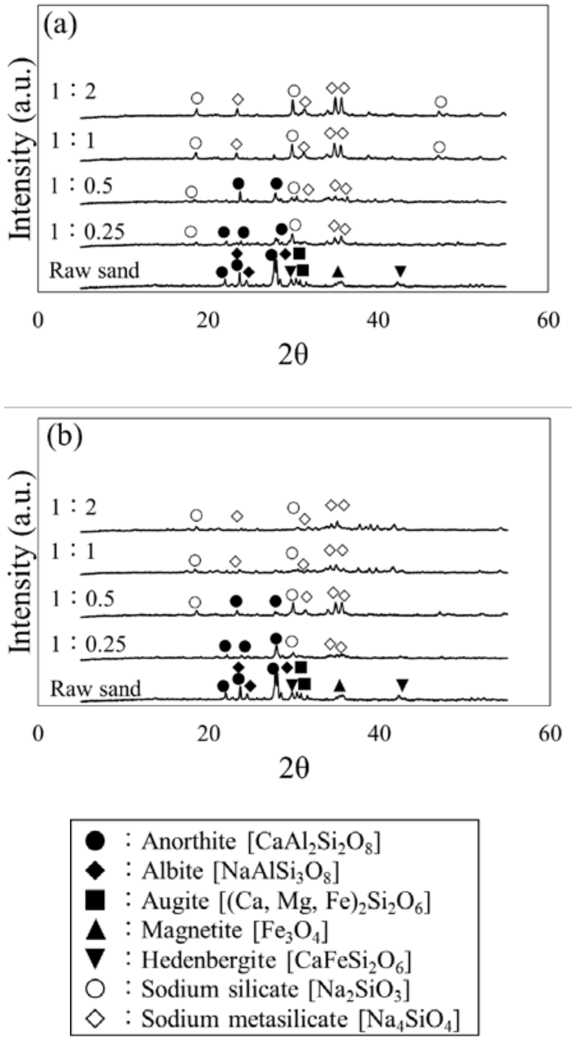

Fig. 1 The XRD patterns of the fused sand prepared by changing the mixing ratio in (a) air and (b) vacuum

The solubility of each element dissolved from the fused sand prepared by changing the mixing ratio in (a) air and (b) vacuum are shown in Fig. 2. In both air and vacuum, as the amount of sodium hydroxide was increased, the solubilities of all elements increased and became almost constant above a mixing ratio of 1: 1. It is considered that almost all elements in the sand was converted into soluble phases by alkali fusion in air and vacuum.

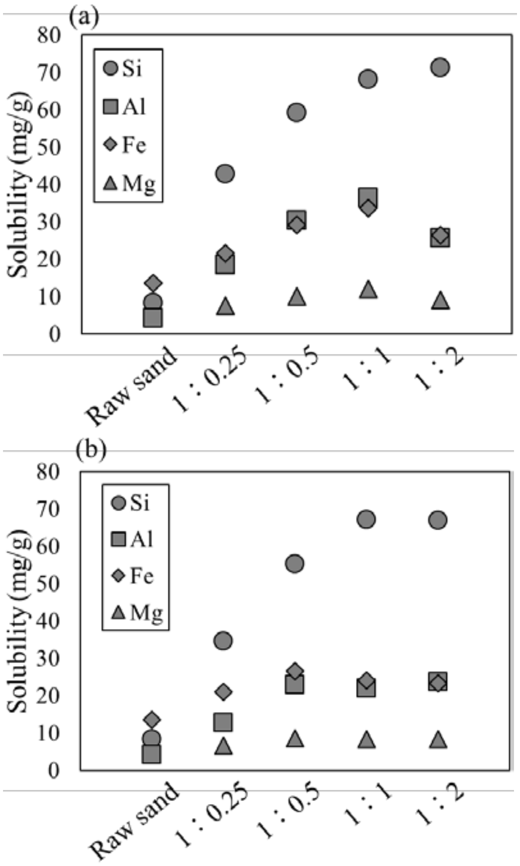

Fig. 2 The solubility of each element dissolved from the fused sand prepared by changing the mixing ratio in (a) air and (b) vacuum

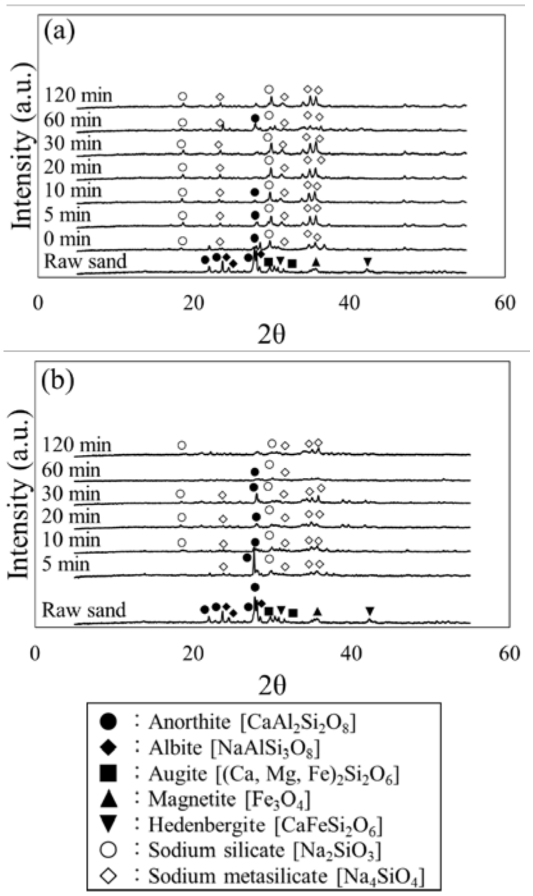

Fig. 3 The XRD patterns of the fused sand prepared by changing the heat time in (a) air and (b) vacuum

3.1.2 Heating Time

The XRD patterns of the fused sand prepared by changing the heating time in (a) air and (b) vacuum are shown in Fig. 3. The fused sand was prepared at the ratio of raw sand: sodium hydroxide = 1: 0.5 at 500 ° C. In the air, as the heating time increased, the peaks of minerals observed in the sand disappeared, and the peak of sodium silicate was confirmed. In the vacuum, as the heating time became longer, the mineral peak observed in the sand disappeared except anorthite, the peaks of anorthite gradually decrease, and the sodium silicate peak was confirmed at more than 5 min.

The solubilities of each element dissolved from the fused sand prepared by changing the heating time in (a) air and (b) vacuum are shown in Fig.4. The solubilities of Si and Al rose and became constant in 10 min, in both air and vacuum. The soluble amounts of Si and Al from the fused sand in air are higher than those in vacuum, which would be caused by remaining anorthite. There was no change in the dissolved amount for Fe, Mg and Ca of all samples.

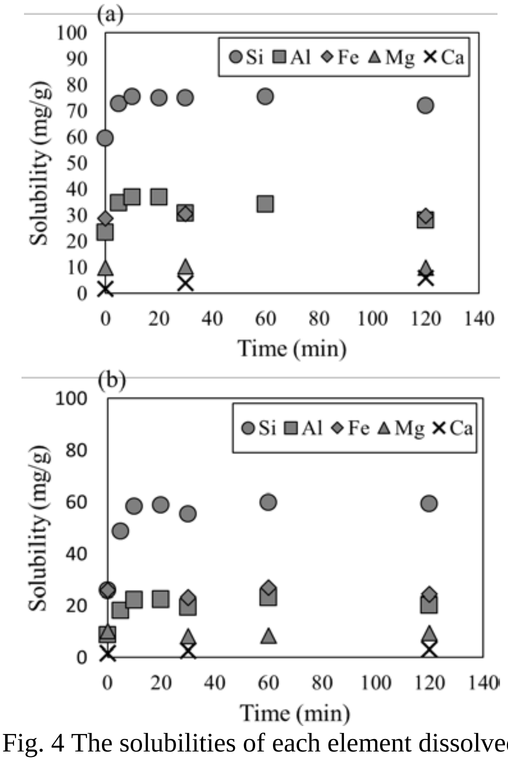

Fig. 4 The solubilities of each element dissolved from the fused sand prepared by changing the heating time in (a) air and (b) vacuum

3.1.3 Heating temperature

The XRD patterns of the fused sand prepared by changing the heating temperature in (a) air and (b) vacuum are shown in Fig. 5. The fused sand was prepared at the ratio of raw sand: sodium hydroxide = 1: 0.5 for 60 minutes. In both air and vacuum, the mineral peaks in the sand disappeared as the heating temperature was raised, and the peaks of sodium silicate were confirmed.

The solubilities of each element dissolved from the fused sand prepared by changing the heating temperature in (a) air and (b) vacuum are shown in Fig. 6. The solubilities of each element increased with increasing the heating temperature in both air and vacuum. At 300 °C, in the air, the fused sands have quadruple higher solubility than that of the raw sand, while in the vacuum, the fused sands have about double higher solubilities than that of the raw sand.

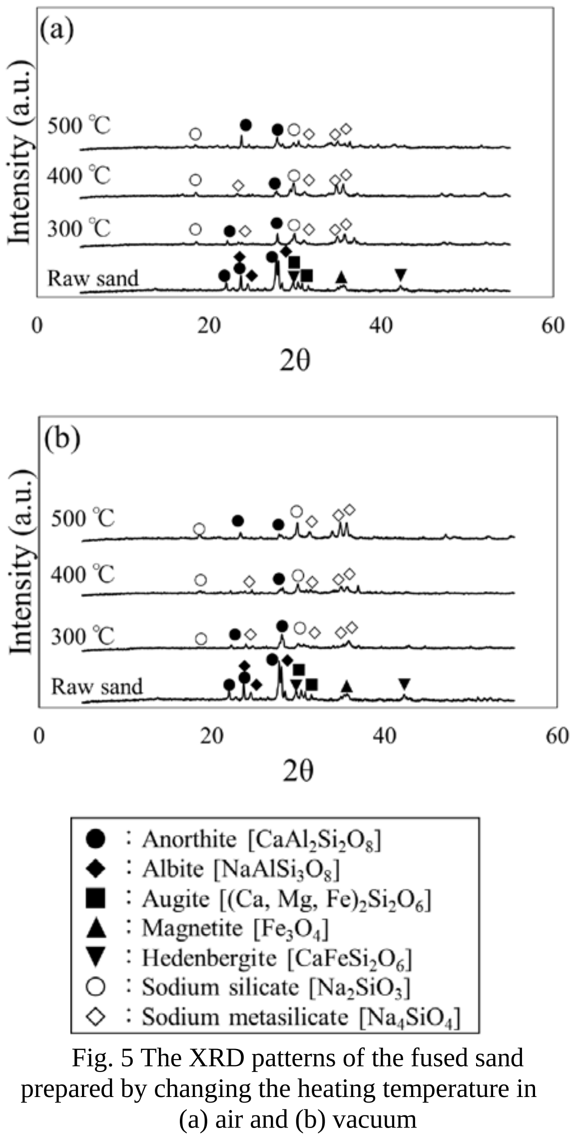

Fig. 5 The XRD patterns of the fused sand prepared by changing the heating temperature in (a) air and (b) vacuum

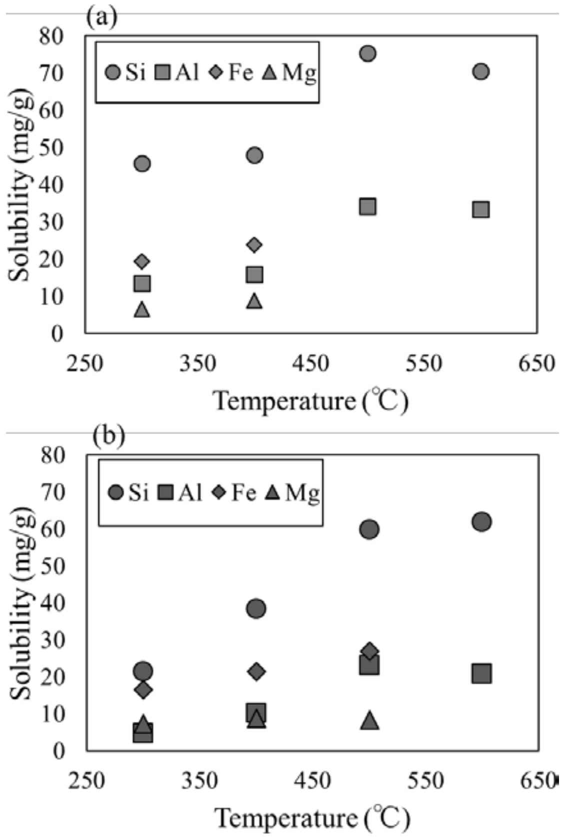

Fig. 6 The solubilities of each element dissolved from the fused sand prepared by changing the heating temperature in (a) air and (b) vacuum

From these results, it is possible to convert the sand into a soluble material including sodium silicate using alkali fusion in vacuum atmosphere, and a high soluble material can be prepared when the mixing ratio is 1:1, heating time is 10 min, and heating temperature is 500 ºC.

3.2 Preparation of Geopolymer Cement

3.2.1 Changing mixing ratio

The photograph of the product after 1 and 7 days from (a) fused sand in air and (b) fused sand in vacuum is shown in Fig. 7. After 1 day, hardened product was confirmed using the fused sand in the air, while the products from the fused sand in vacuum are slightly soft. After 7 days, all the products were hardened.

The result of the strength test for the product prepared from (a) fused sand in air and (b) fused sand in vacuum is shown in Fig. 8. It was confirmed that the strength increased as the curing time was prolonged in all the products. The product with the highest strength was obtained using the fused sand prepared in the air atmosphere at the mixing ratio of 2: 1: 2.

The product using the fused sand prepared in a vacuum atmosphere at a mixing ratio of 2: 1: 2 had the highest strength (120 N) among the product from fused sand in vacuum.

Portland cement indicate higher strength (500 N) than the obtained products. The highest strength of the obtained product is 200 N, which is less than half as strong as the Portland cement.

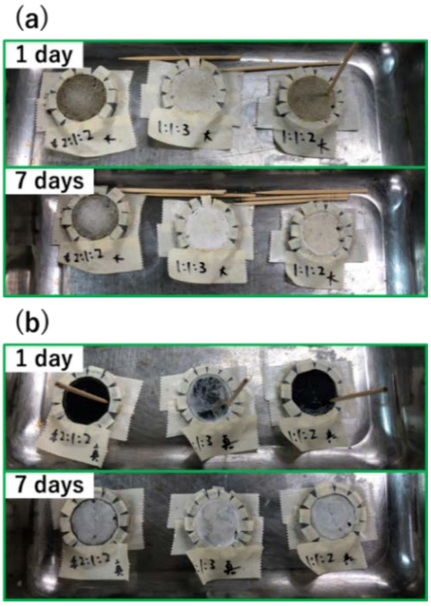

Fig.7 The photograph of the product after 1 and 7 days from (a) fused sand in air and (b) fused sand in vacuum

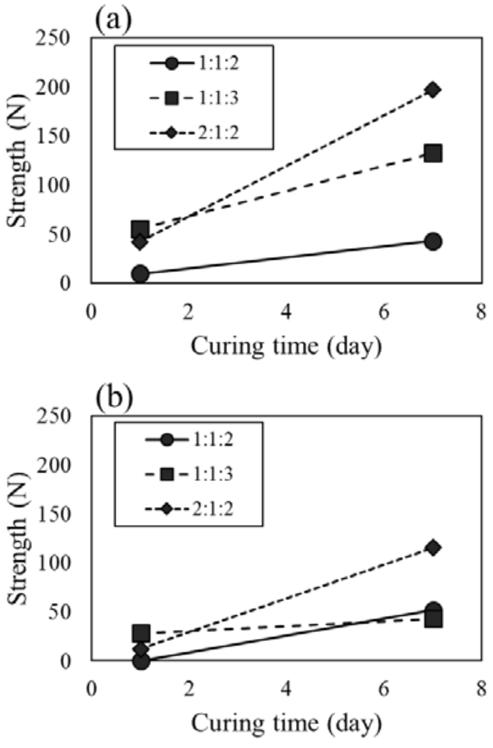

Fig. 8 The result of the strength test for the product from (a) fused sand in air and (b) fused sand in vacuum

3.2.2 Changing temperature and atmosphere

Fig. 9 shows the strength of the geopolymer cement produced by changing the heating atmosphere and the heating temperature. The geopolymer cement was prepared with raw sand: water: fused sand = 2: 1: 2. It was confirmed that the geopolymer cement prepared in the atmosphere had a strength of 300 N in the sample heated at 106 °C in both air and vacuum atmosphere. It was confirmed that the geopolymer cement using the melt prepared in a vacuum atmosphere was a geopolymer cement heated at 106 °C in a vacuum atmosphere and had a strength of 200 N.

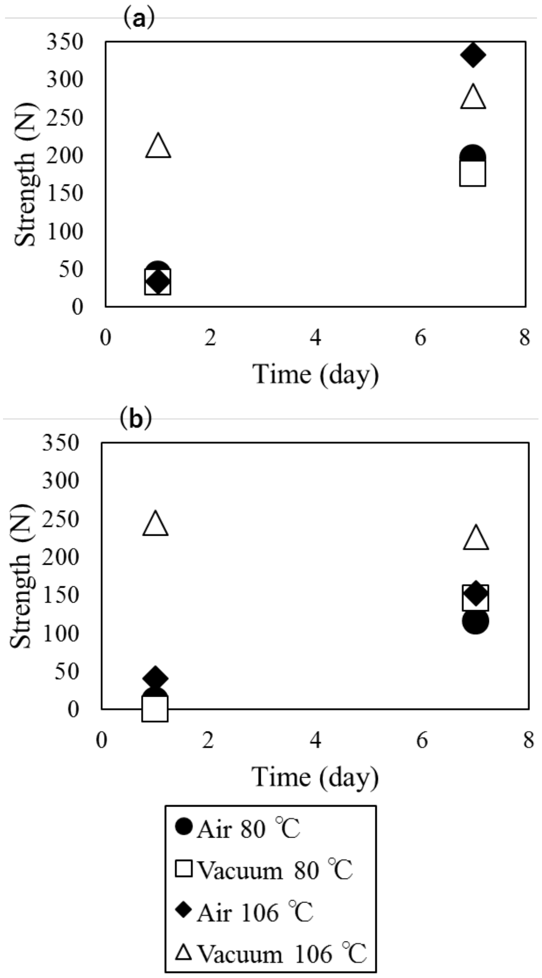

Fig. 9 The result of the strength of the geopolymer cement produced by changing the heating atmosphere and the heating temperature from (a) fused sand in air and (b) fused sand in vacuum

3.2.3 Adaptation to the moon

Figure 10 shows the strength of the geopolymer cement after heating and cooling. The geopolymer cement was prepared on a simulated lunar surface with raw sand : water : fused sand (vacuum) mixture ratio of 2 : 1 : 2, a heating temperature of 106 ° C, and a heating time of 7 days. Heating and cooling reduced the strength by about 20% compared to untreated geopolymer cement.

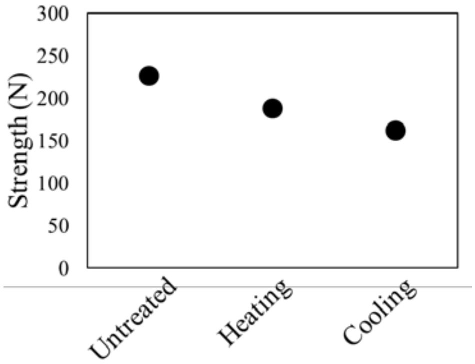

Fig. 10 The result of the strength test for the product

4. CONCLUSION

An attempt was made to prepare geopolymer cement by alkali fusion of simulated lunar rock sand with sodium hydroxide. Alkali fusion with sodium hydroxide allows conversion to a highly soluble fused sand even under vacuum. It was found that high soluble fused sand can be produced with mixing ratio of 1: 1, heating temperature of 500 ° C, and heating time of 10 min.

It was successful in making a hardened geopolymer cement by mixing the raw sand, water and fused sand. The strength of 200 N was confirmed with the geopolymer cement prepared at a mixing ratio of 2:1:2, using the fused sand prepared in the air atmosphere. Since this strength of the obtained product is about half the strength of the Portland cement, it is necessary to improve the strength. The highest strength of the geopolymer cement using the fused sand prepared in the air was confirmed at 106 °C in the air. The highest strength of the geopolymer cement using the fused sand prepared in a vacuum was confirmed at 106 ° C in a vacuum.

5. ACKNOWLEDGMENTS

This work was supported by the Grant Aid of Japan Crushed Stone Association. The author would like to thank to you here.

6. REFERENCES

[1] Davis G., C Montes. and Eklund S., Preparation of lunar regolith based geopolymer cement under heat and vacuum, Advances in Space Research, 59, 2017, pp. 1872 – 1885.

[2] Onishi S., Wajima T., Imai T. and Susumu S., Alkali Fusion Process of Waste Stone Dust to Synthesize Faujasite Using Rotaky Kiln, Mechanics, Materials Science & Engineering Journal, Vol. 9, No. 1, 2017, pp. 287-292.

[3] Wajima T., Alkali Fusion Synthesis of Zeolitic Materials from Waste Dehydrated Cake Discharged from Recycling of Construction Waste Soil, Natural Resources, Vol. 8, 2017, pp. 300-305.

[4] Wajima T., Synthesis of zeolite from blast furnace slag using alkali fusion with addition of EDTA, Advanced Materials Research, pp. 1044-1045, p.p. 124-127.

[5] T Wajima. and Munakata K., Material conversion from waste sandstone cake into cation exchanger using alkali fusion, Ceramics International, Vol. 38, No. 2, 2012, pp. 1741–1744.

[6] Wajima T., Munakata K. and Ikegami Y., Conversion of Waste Sandstone Cake into Crystalline Zeolite X Using Alkali Fusion, Materials Transactions, Vol. 51, No. 5, 2010, pp. 849-854.

[7] Wajima T., Yoshizuka K., Hirai T. and Ikegami Y., Synthesis of Zeolite X from Waste Sandstone Cake Using Alkali Fusion Method, Materials Transactions, Vol. 49, No. 3, 2008, pp. 612-618.

[8] Sakamoto K. and Wajima T., Preparation of Geopolymer Cement from Crushed Stone By-Product Using Alkali Fusion, International Journal of GEOMATE, Vol.17, Issue 63, 2019, pp.17-22.

[9] Melkon T., Gunther M. and Stefan K. H., Relation of water adsorption capacities of zeolites with their structural properties, Microporous and Mesoporous Materials 264, 2018, pp. 70–75.

[10] Habbib G., Oliver S., Yvan M. and Philippe K., The Reconstruction of Natural Zeolites, Kluwer Academic Publishers, Dordrecht, pp. 1-20.