

Adsorption of Carbon Dioxide on Shirasu-based Materials

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This work investigated adsorption of carbon dioxide on Shirasu-based materials with various pretreatment procedures. A Shirasu-based material pretreated via calcination procedure followed by treatment in aqueous ammonia solution adsorbed significantly high amount of carbon dioxide compared with pristine Shirasu-based material and the Shirasu-based material

pretreated via only solution treatment in aqueous ammonia solution. The ability improved through addition of appropriate amount of water before the adsorption process of carbon dioxide, and exhibited comparative adsorption amount of carbon dioxide with high performance materials previously reported.

Introduction

Since the industrial revolution, gigantic amount of materials such as foods, fuels, etc. has been consumed accompanied with human activities. Along with those consumptions, various chemical compounds such as carbon dioxide has been excessively emitted and accumulated in the global environment because excess amount of the compounds are not circularly converted via the sustainable natural process such as photosynthesis process. In these compounds, carbon dioxide has been regarded as a greenhouse gas that has burdened the global environment. Adsorption processes of carbon dioxide in solution^[1–5] and on solid materials^[6–8] has been proposed as promising processes for storing carbon dioxide excessively emitted into the atmospheric environment. In the various processes via adsorption, porous solid materials including oxide ceramics such as aluminosilicate including zeolites,^[9–22] mesoporous silicas,^[23–30] metal oxides,^[31–33] carbon-based materials,^[34–38] and metal-organic-frameworks (MOFs)^[39–41] have attracted much attention for cyclic adsorption uses in low-cost simple adsorption processes of carbon dioxide.

In the present study, we reported adsorption of carbon dioxide on Shirasu-based materials for the first time. Shirasu is fine-grained pumice and volcanic ash, distributed as a thick stratum throughout southern Kyushu, Japan.^[42] Most Shirasu is now disposed as solid waste to landfills.^[43] The application of volcanic sediment as a raw material of adsorbents has some advantages in reducing the amount of solid waste and the cost for the treatment. The materials are also expected to exhibit similar adsorption functions as zeolites because Shirasu-based

materials mainly included silica and alumina which were also included in zeolites. It has been reported that the Shirasu-based materials can adsorb ions^[44–46] and compounds.^[47,48] On the other hands, surface modification of Shirasu-based materials were effective ways to improve their adsorption capacities of the chemical species.^[44,45,47,48] We have also identified the effect of surface modification of a Shirasu-based material via various pretreatment procedures (solution treatment and calcination) on improving its surface acidity and promoting a chemical reaction in the presence of acids.^[49] While, it has been reported that adsorption capacities of carbon dioxide of zeolites were improved by increasing their acidity.^[9,11] From these points of view, this work investigated the influence of various pretreatment procedures on the adsorption ability of carbon dioxide of a Shirasu-based material.

Results and Discussion

Table 1 lists composition of main components in a commercial Shirasu material, SSM, treated with various procedure evaluated from the results of EDS analyses and XPS spectra. From the table, bulk ratios of silicon to aluminium depended on the samples, while, surface ratios of silicon to aluminium decreased with additional procedures such as treatment in aqueous ammonia solution and calcination. The results suggest that surface compositions can be significantly controlled by the procedure in aqueous ammonia solution following the calcination procedure probably because of the effects of solution etching and calcination. For all the samples, we have previously reported that BET specific surface areas of SSM, SSMNH₃, and SSMcalNH₃ were

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Table 1. Bulk and surface compositions of SSM samples treated with various procedures.

	Si/Al _{bulk} ^[a]	Si/Al _{surface} ^[b]
SSM	1.7	5.0
SSMNH ₃	3.3	2.2
SSMcalNH ₃	2.0	1.1

[a] Calculated from the results of EDS. [b] Calculated from the results of XPS.

8.8, 7.7, and 6.1 m^2g^{-1} , respectively, and that all the samples consisted of rock-like particles with the size up to about 100 μm accompanied with irregular-shaped particles with the size around 1 μm from the SEM images.^[49] These results suggest that specific surface area and morphology of SSM did not significantly change through the solution treatment and calcination. Figure 1 shows nitrogen sorption isotherms (A) and pore size distributions (B) of the samples estimated from the result of nitrogen sorption measurements to obtain further information of morphological properties. All the samples exhibited type IV isotherms and the

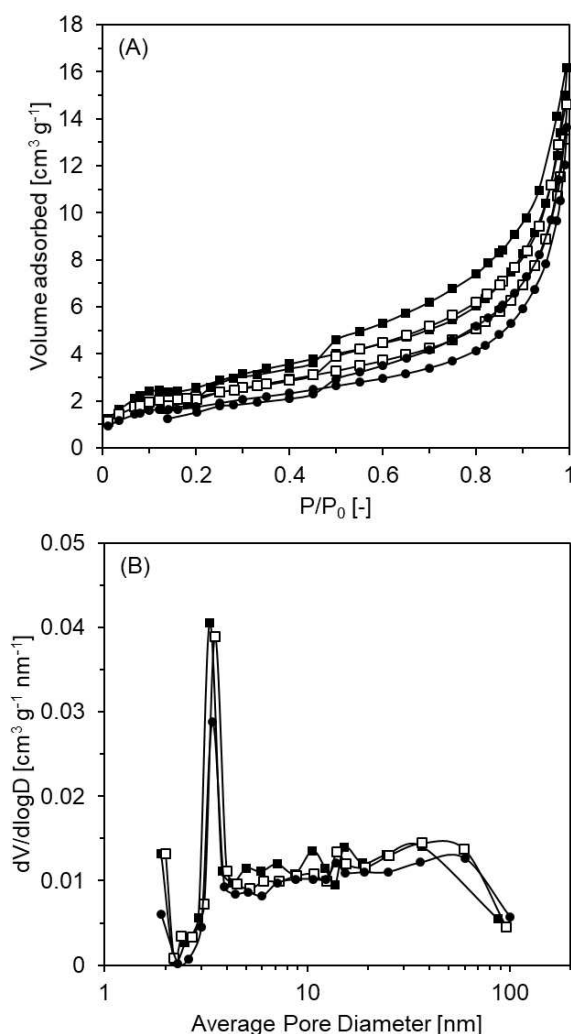


Figure 1. (A) nitrogen sorption isotherms and (B) pore size distributions of (a) SSM, (b) SSMNH₃, and (c) SSMcalNH₃.

Table 2. Textural properties of SSM samples treated with various procedures.		
	Pore Volume [cm^3g^{-1}] ^[a]	Average Pore Diameter [nm] ^[a]
SSM	0.024	8.8
SSMNH ₃	0.022	9.2
SSMcalNH ₃	0.021	10.4

[a] Calculated from the nitrogen desorption branch using the BJH method.

hysteresis loops were type H4 which suggested the existence of mesopores in all the samples,^[50] while, the sorption amounts decreased in order to SSM > SSMNH₃ > SSMcalNH₃. The isotherms reflect in the pore size distributions (Figure 1(B)). From the figure, all the samples exhibited the similar distributions except for the dV/dlogD values of the main peak at the average pore diameter of around 3.5 nm. The peak value of SSMcalNH₃ was low compared with SSM and SSMNH₃. Table 2 lists pore volumes and average pore diameters of the samples calculated from the result in Figure 2. From the table, pore volumes of all the samples were same levels, while, average pore diameter of SSMcalNH₃ was larger than those of SSM and SSMNH₃. The results suggested that all the samples possessed similar pore analyzed by DRIFT spectra and TG analyses. Figure 2 displayed DRIFT spectra of the SSM-based samples with various pretreatment procedures before and

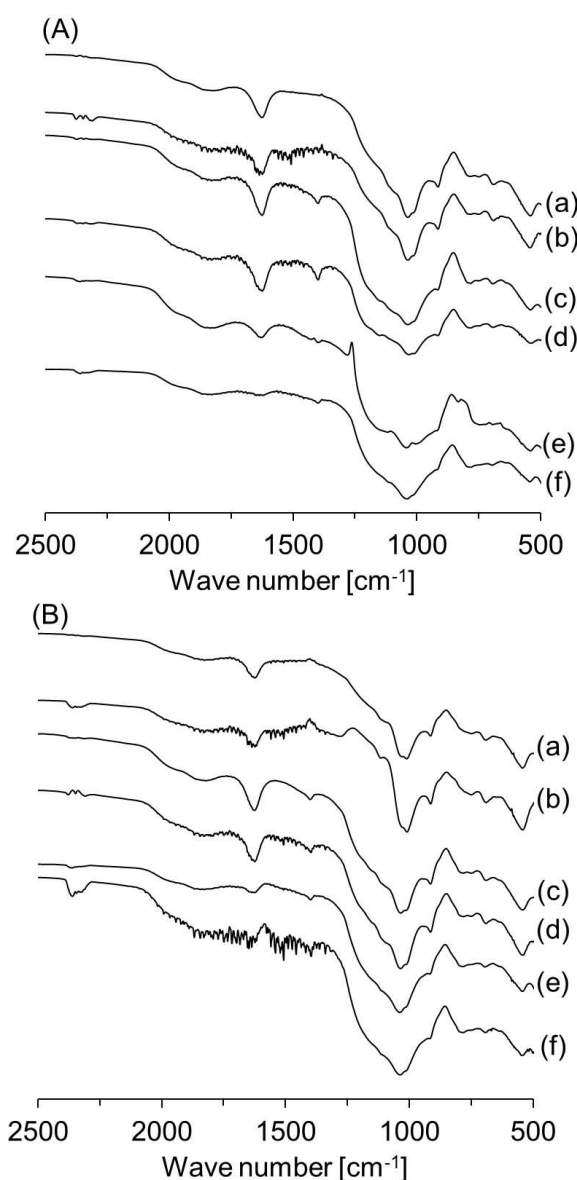


Figure 2. DRIFT spectra of the samples treated without (A) and with 30 $\mu\text{L-H}_2\text{O g-sample}^{-1}$ (B) before (a, c, e) and after (b, d, f) adsorption of carbon dioxide. Samples: (a, b) SSM, (c, d) SSMNH₃, (e, f) SSMcalNH₃. Adsorption conditions: 293 K, 1 MPa CO₂, 1 h.

after adsorption of carbon dioxide. The observable band centered at ca 2348 cm^{-1} assigned as adsorbed carbon dioxide

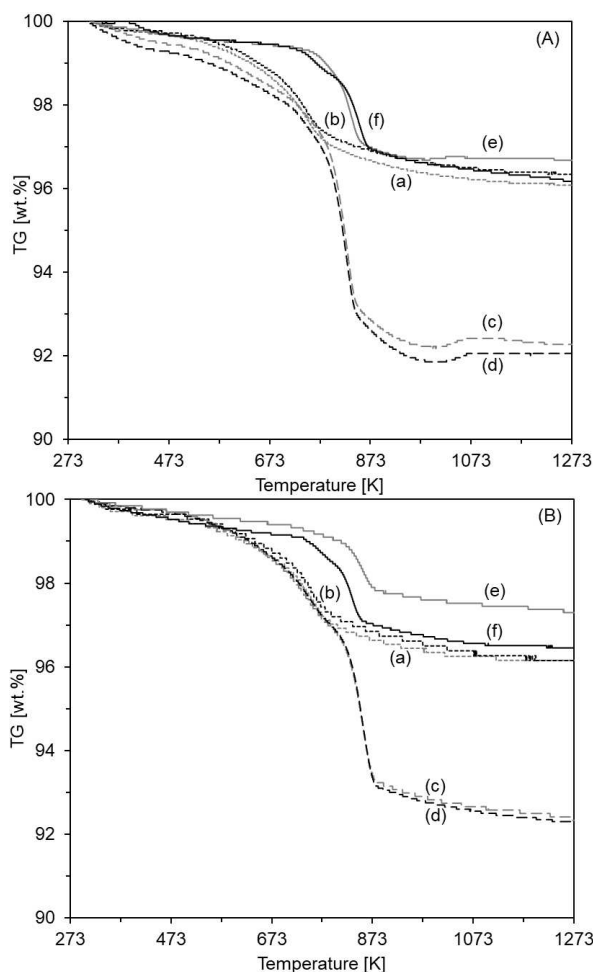


Figure 3. TG curves of the samples treated (A) without and (B) with $30\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$. (a, b) SSM, (c, d) SSMNH₃, (e, f) SSMcalNH₃.

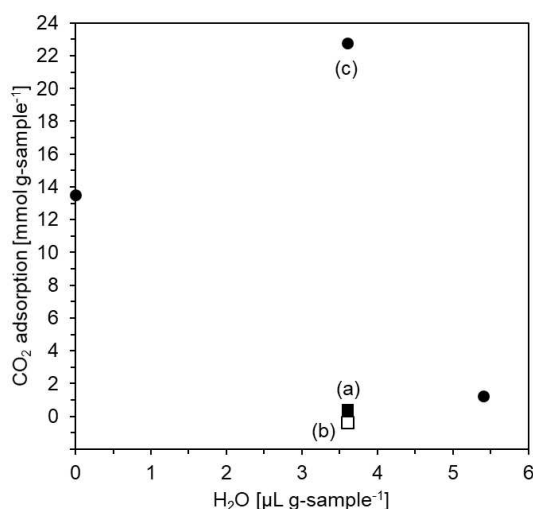


Figure 4. Influence of added water amount on amount of adsorbed carbon dioxide on Shirasu-based materials with various pretreatment procedures. (a) SSM, (b) SSMNH₃, and (c) SSMcalNH₃.

molecules^[15,19,27] was confirmed in the spectra after adsorption procedure of carbon dioxide, while, the band intensity significantly depended on the samples and the treatment with water just before adsorption of carbon dioxide. Especially, band assigned as the adsorbed carbon dioxide was clearly identified in the spectrum of the samples treated with $30\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$ of water, indicating that the sample physically adsorbed high amount of carbon dioxide. Figure 3 showed TG curves of the samples treated without and with $30\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$. The slight differences of the weight losses before and after the adsorption of carbon dioxide were observed in SSM and SSMNH₃ treated without and with water (Figure 3 (A) and (B)), while, SSMcalNH₃ treated with water exhibited large difference of weight loss before and after the adsorption procedure with carbon dioxide. The result indicates that SSMcalNH₃ treated with water exhibited the highest adsorption capacity of carbon dioxide in all the samples treated without and with $30\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$. Figure 4 displayed the adsorption amount of carbon dioxide on SSMcalNH₃ treated with various amount of water before adsorption of carbon dioxide. The adsorption amount was quantified from the results of TG analyses of the samples before and after the adsorption of carbon dioxide. The sample treated with $30\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$ showed higher adsorption amount of carbon dioxide than that of the sample without water treatment. From the results in Figure 1e, the adsorption band centered at ca. 1630 cm^{-1} assigned as physisorbed water molecules^[15,51,52] was observed in both the spectra, while, the band intensity in the spectrum of the sample treated with water was higher than that in the spectrum of the sample without water treatment. The intensity ratios of the bands of water to the band centered at ca. 1040 cm^{-1} assigned as silicon oxide^[21,53,54] of the samples prepared treated without and with water were 0.090 and 0.026, respectively. The results indicate that residual adsorbed water decreased with water treatment before adsorption of carbon dioxide. The tendency was confirmed in comparison to the adsorption capacities of SSM and SSMNH₃, and SSMcalNH₃ showed significantly high adsorption amount of carbon dioxide in comparison to SSM and SSMNH₃. The intensity ratios of the band assigned as water to that of the band assigned as silica in the spectra of SSM and SSMNH₃ were 0.175 and 0.141, respectively, indicating that the amount of residual adsorbed water of SSM and SSMNH₃ were high after water treatment and the adsorption amount of carbon dioxide on the samples were significantly low in comparison to that of SSMcalNH₃ as shown in Figure 2. On the other hand, the adsorption amount of carbon dioxide of the SSMcalNH₃ sample treated with $45\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$ was also evaluated, and the amount drastically decreased in comparison to SSMcalNH₃ treated without and with $30\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$. From the result of DRIFT spectrum of the sample treated with $45\text{ }\mu\text{L-H}_2\text{O g-sample}^{-1}$, the intensity ratio of the band assigned as water to the band assigned as silica was 0.006. From the results of DRIFT spectra and adsorption of carbon dioxide, the sample treated with appropriate amount of residual adsorbed water showed high adsorption capacity of carbon dioxide. Promoting effect of preadsorbed water has been reported on some silica-based materials,^[11,19,21–23,27] and adsorption of carbon dioxide on the materials was improved by the

existence of appropriate amount of water molecules. The zeolites, which contain similar components as Shirasu materials, include both Brønsted acid sites and Lewis acid sites. The Lewis acid sites are formed with octahedrally coordinated nonframework aluminum species, and can be transformed to Brønsted acid sites via coordinating water molecules.^[11] It has been reported that Brønsted acid sites contributed to enhance the physisorption of carbon dioxide.^[9,11] From these points of view, it is suggested that the adsorption capacity of SSMcalNH₃ can be improved by effect of adding appropriate amount of water (30 $\mu\text{L-H}_2\text{O g-sample}^{-1}$) to transform the Lewis acid sites in the sample to the Brønsted acid sites. On the other hand, it has been also reported that water molecules can also compete with carbon dioxide to adsorb on the sites of the zeolites. The adsorption capacity of SSMcalNH₃ treated with 45 $\mu\text{L-H}_2\text{O g-sample}^{-1}$ was low probably because of blockage of the sites with excessively adsorbed water molecules. The dependence of

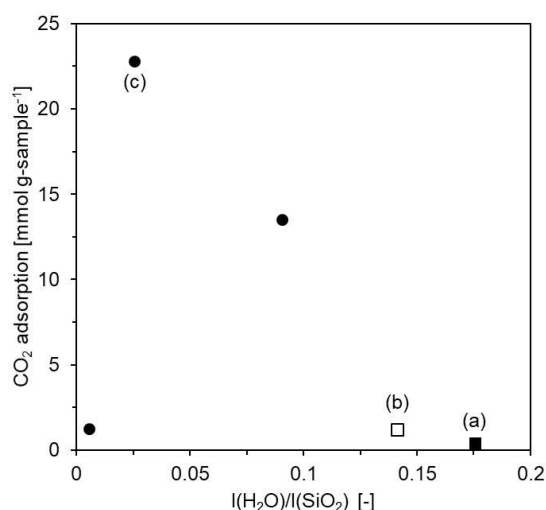


Figure 5. Dependence of adsorption of carbon dioxide against ratios of the band intensity assigned as water to silicon. (a) SSM, (b) SSMNH₃, and (c) SSMcalNH₃.

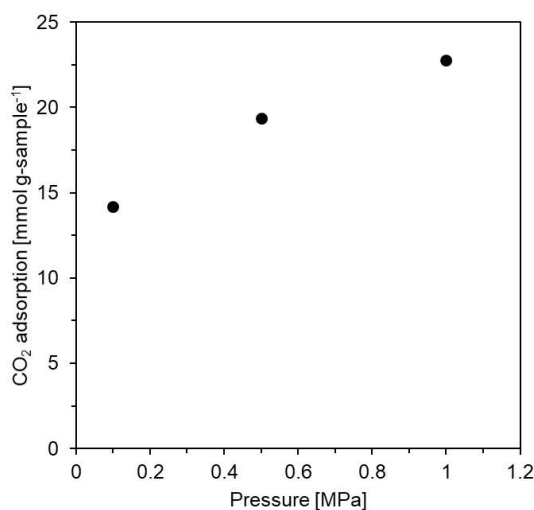


Figure 6. Dependence of adsorption amount of carbon dioxide of SSMcalNH₃ treated with 30 $\mu\text{L-H}_2\text{O g-sample}^{-1}$ on adsorption pressure.

adsorption of carbon dioxide on the amount of water on the samples in the present study reflected the tendency of the results in the previous studies. Figure 5 exhibited dependence of adsorption amount of carbon dioxide of the samples on the band intensity ratios of water to silicon. From the figure, the adsorption amount increased with increasing the intensity ratios. Figure 6 displayed dependence of adsorption amount of carbon dioxide of SSMcalNH₃ treated with 30 $\mu\text{L-H}_2\text{O g-sample}^{-1}$ on the adsorption pressure. As shown in this figure, the adsorption amount of carbon dioxide increased with increasing adsorption pressure, while, the sample adsorbed carbon dioxide even at ambient pressure. The dependence of adsorption pressure is characteristic of physisorption processes of carbon dioxide as the processes on many typical adsorbents such as zeolites^[10] and functionalized mesoporous silica.^[24] From the results of DRIFT spectra in Figure 2, the low intensity band at ca1400 cm^{-1} assigned as ammonium ion was observed, while, the band was also observed in the spectra of SSMNH₃ which exhibited low adsorption capacity of carbon dioxide. The results suggested that the adsorbed basic ammonium ion showed no effect on promoting adsorption of carbon dioxide. The adsorption amount is much higher than those of zeolitic materials (ex) 13X: 6.9 $\text{mol kg-sample}^{-1}$ (15 bar)^[9–22] and comparative to the amount on high performance MOF materials (ex) MOF-177: 32 $\text{mmol g-sample}^{-1}$ (4.2 MPa)).^[39–41]

Conclusions

The present work investigated influence of various pretreatment procedures of a Shirasu-based material and water addition on the material on its adsorption capacity of carbon dioxide. A Shirasu-based material, SSM, were treated in aqueous ammonia solution and calcined following the treatment in aqueous ammonia solution. Adsorption amount of carbon dioxide depended on the procedures, the amount of residual water molecule significantly influenced on the adsorption amount. From the result of adsorption measurements of carbon dioxide on the samples treated with various amount of water, the sample with appropriate amount of the residual water molecules exhibited high adsorption amount of carbon dioxide, which comparative adsorption amount of carbon dioxide to reported high performance materials even at ambient pressure.

Experimental Section

Sample preparation

Commercial Shirasu sample, SSM, was provided from Takachiho-Shirasu Co. Ltd. The samples were treated in aqueous ammonia solution (20 mL (0.8 g of the sample treating), 28.0~30.0%, Kanto Chem. Co.). One of the ammonia-treated samples were pre-calcined at 723 K in air for 2 h. These treated samples were denoted SSMNH₃ and SSMcalNH₃.

Characterizations

Elemental analyses of the samples were conducted with a XFlash Mini SVE (Brucker) for energy dispersive X-ray spectroscopic (EDS) analysis. The textural properties of the samples were analysed by nitrogen sorption measurements at 77 K with a Model ASAP 2010MC (Micromeritics Instrument Co.). For analyses of the surface compositions, X-ray photoelectron spectra (XPS) were recorded with an ESCA3400 spectrometer (Kratos Analytical Ltd.) equipped with a Mg K α X-ray exciting source (1253.6 eV) operating at 10 kV and 20 mA. The binding energies (BE) referred to the C 1s peak at 285.0 eV. The DRIFT spectra of the samples were recorded using a IRTSprit Fourier transform infrared spectrophotometer (Shimadzu Co. Ltd.) scanned from 5000 to 400 cm⁻¹ with a resolution of 4 cm⁻¹.

Assessment for adsorption capacity of carbon dioxide

For assessing adsorption capacity of carbon dioxide, the sample (0.1 g) was put into the closed reactor and was treated under 0.1–1 MPa of carbon dioxide atmosphere at 293 K for 1 h. Adsorption amount of carbon dioxide on the samples were estimated from the results of thermal gravimetric analysis (TGA) with an DTG-60 (Shimadzu Co. Ltd.) in nitrogen flow (20 mL min⁻¹) before and after the adsorption process of carbon dioxide,

Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords: Shirasu-based materials · adsorption of carbon dioxide · addition of water

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