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Conversion of CO_2 to Formic Acid Using Silicon Sludge as a Reducing Agent

Jincheng Wu†1), Yusuke Tanimura†1), Danyang Liu†1), Shingo Hasegawa†1),
Kousuke Arata†2), Ryosuke Takemura†2), Kazuo Namba†2), and Ken Motokura*†1)

†1) Dept. of Chemistry and Life Science, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, Yokohama 240-8501, JAPAN
†2) Electric Power Development Co., Ltd., 1 Yanagasaki-machi, Wakamatsu-ku, Kitakyushu 808-0111, JAPAN

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Utilizing Si sludge from wafer-production industries for synthesizing value-added chemicals using CO_2 facilitates both the recycling of waste Si and the utilization of CO_2 as a carbon resource. In this study, the reduction of CO_2 to produce formic acid using Si sludge as a reducing agent is reported. Tetrabutylammonium fluoride effectively promotes the reduction. This approach is suitable not only for pure CO_2 but also for the CO_2 present in the exhaust gas from a thermal power plant. Reaction parameters such as CO_2 pressure, temperature, water content, and amount of silicon sludge were optimized. Analyses of the Si sludge before and after the CO_2 reduction reaction were performed using XPS and XRD, which indicated the conversion of the Si^0 phase to SiO_2 in the Si sludge. This is an environmentally friendly and possibly cost-effective approach for formic acid synthesis since CO_2 is converted to formic acid by Si sludge.

Keywords
Silicon sludge, Carbon dioxide, Formic acid, Reduction, Fluoride catalyst

1. Introduction

Silicon is widely used in the semiconductor industry. Methods to recycle Si have been extensively explored because of its importance. Chemical recovery methods, such as acid/base dissolution and hydrometallurgical processes, are employed to extract highly pure Si from end-of-life solar cells1)~3). Recently, the use of waste Si as a reducing agent has gained considerable attention. This approach has facilitated the conversion of fractured silicon wafers and iron oxide into ferrosilicon alloys4). Methods to recycle Si sludges obtained while producing silicon materials, such as silicon wafers, are also highly desirable.

CO_2, being a major greenhouse gas, significantly influences global climate change. Hence, efficient methods for its capture and conversion are required5)~7). CO_2 transformation using electrocatalytic, photocatalytic, and chemical reduction techniques have been investigated. Currently, CO_2 chemical reduction methods using hydrosilanes and related compounds as reducing agents are widely employed, owing to their high efficiency and controllability in CO_2 conversion process (**Fig. 1**)8)~18). Das et al. explored the utilization of hydrosilanes as reducing agents and examined catalysts such as metallic, organocatalysts, and multiphase catalysts to promote the hydrosilylation of CO_2 for the synthesis of valuable C1 chemicals such as formic acid, methanol, and methane19). Parkin et al. investigated the application of hydrosilane in the reduction of CO_2 and explored the conversion of carbon dioxide to valuable C1 chemicals, such as siloxyl formate and methoxy derivatives, under the catalytic action of metal complexes20). In addition, Si-based materials have been employed as CO_2 reducing agents21)~23). Ozin et al. investigated the photothermal catalytic properties of hydride-terminated silicon nanocrystals (ncSi:H) for CO_2-to-CO conversion, highlighting the potential of Si as an efficient and cost-effective CO reduction material21). Dasog et al. further explored the direct conversion of CO_2 to methanol using porous silicon nanoparticles (Si-NPs) without the need for additional catalysts23). In our previous study, fluoride-catalyzed

$$R_3Si\text{-}H + CO_2 \xrightarrow{\text{catalyst}} HCOOSiR_3$$

Fig. 1 Reduction of CO_2 with Hydrosilane

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* To whom correspondence should be addressed.
* E-mail: motokura-ken-xw@ynu.ac.jp

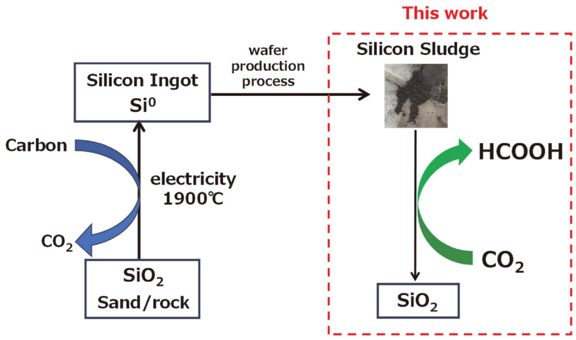
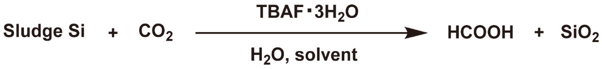


Fig. 2 Waste Si Used as a Reducing Agent of CO_2 in the Si Cycle



$$\text{Sludge Si} + CO_2 \xrightarrow[H_2O,\ \text{solvent}]{TBAF \cdot 3H_2O} HCOOH + SiO_2$$

Fig. 3 Production of Formic Acid from Silicon Sludge

CO_2 reduction reactions using hydrosilane and powdered silicon were investigated, and valuable chemicals such as formic acid and methanol were synthesized[24)~34)].

Using Si waste from industries (such as silicon wafer production industries) in the reduction of CO_2, would facilitate the recycling of waste Si as well as the valuable utilization of CO_2. In addition, zero-valent silicon was prepared from sand (SiO_2) with large amounts of electricity consumption and CO_2 emissions. The use of waste Si as a reducing agent of CO_2 refers to the recovery of the emitted CO_2 in the Si material cycle (**Fig. 2**).

In this study, formic acid was successfully synthesized from CO_2 using Si sludge recovered from Si wafer production (**Fig. 3**). Key reaction parameters, such as CO_2 pressure, temperature, water content, and silicon sludge loading, were systematically optimized to enhance the conversion efficiency. Pure CO_2 and the CO_2 released from a thermal power plant were used. The silicon sludge before and after the reduction of CO_2 was analyzed using X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD) to assess its composition, oxidation state, and crystal structure. In particular, the impact of SiC impurities was considered. This research not only addresses the problem of Si waste accumulation but also presents an eco-friendly strategy for CO_2 utilization.

2. Experimental

2. 1. Materials

Silicon sludge was generated as a byproduct during wafer preparation. The silicon sludge contains impurities such as Fe(14.9 wt%), Na(9.4), Al(8.3), Cu(6.1), Ca(4.9) K(2.6), Mg(1.5), and other trace metals. Tetrabutylammonium fluoride trihydrate ($TBAF \cdot 3H_2O$, $>$99 %) was purchased from Kanto Chemical Co., Inc. Chloroform-d_1 (98 %D) stabilized with silver foil was purchased from Kanto Chemical Co., Inc. *N*-Methyl-2-pyrrolidone (NMP, dehydrated, $>$99 % pure) was purchased from Kanto Chemical Co., Inc. (used without further purification). Deionized water (H_2O) was prepared in the laboratory. All other materials were purchased from Tokyo Chemical Industry Co., Ltd., Kanto Chemical Co., Inc., and Aldrich Inc. Exhaust gas containing CO_2 (14.0 %CO_2-5.0 %O_2-N_2 bal.) was supplied by the Isogo thermal power plant (coal-fired), Electric Power Development Co., Ltd.

2. 2. Analytical Methods

^{1}H nuclear magnetic resonance (NMR) spectra were acquired in $CDCl_3$ using a JEOL RESONANCE ECA 500 spectrometer (operating at 500 MHz for ^{1}H) or an ECX 400 spectrometer (operating at 400 MHz for ^{1}H). XPS analyses were conducted on a ULVAC-PHI Quantera-SXM system with an Al X-ray source (pass energy = 55.0 eV). The Al Kα X-ray source operated at 50.2 W and 14 kV with a beam size of 200 μm was used, and excess charges on samples were neutralized through argon-ion sputtering. The analysis chamber maintained a working pressure below 1×10^{-7} Pa, with spectra collected in the C 1s, Si 2p. The XPS elemental peaks were referenced to the C 1s position at 284.8 eV. Powder XRD patterns were generated using a Rigaku Ultima IV diffractometer with Cu Kα radiation. Gas phase products were analyzed by gas-chromatography with barrier discharge ionization detector (GC-BID) using Shimadzu GC-2010Plus instrument with a micropacked ST (SHINCARBON) column.

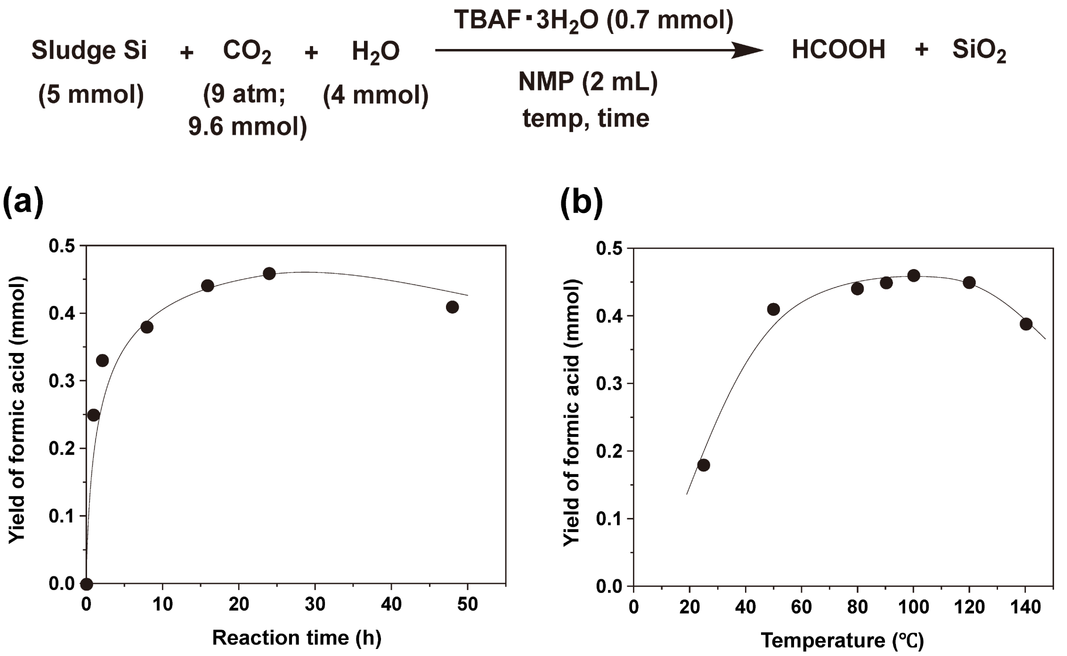
2. 3. Typical Procedure for Fluoride-catalyzed CO_2 Reduction with Si Sludge Powder and H_2O

Stainless steel autoclave was used as the reactor. 0.14 g of powdered Si sludge was placed in the reactor. A mixture of the catalyst ($TBAF \cdot 3H_2O$, 0.70 mmol), solvent (NMP, 2 mL), and a specific amount of deionized water (4 mmol) was prepared in a separate vial and transferred into the reactor. Subsequently, a reaction gas (CO_2 at 0.9 MPa) was introduced. The resulting mixture was vigorously stirred at 100 ℃ for 24 h. The reaction products were identified using ^{1}H NMR spectrometry. The internal standard method was used to determine the yield of formic acid using liquid ^{1}H NMR in $CDCl_3$ (internal standard: 1,3,5-triisopropylbenzene). The quantitative relationship between the ^{1}H NMR peak area of formic acid and that of the internal standard was validated using standard samples. The gas phase products, such as CO and H_2, in the reactor were qualitatively and quantitatively analyzed by GC-BID.

3. Results and Discussion

3. 1. Optimization of Reaction Conditions

TBAF (a homogeneous catalyst in NMP) facilitated



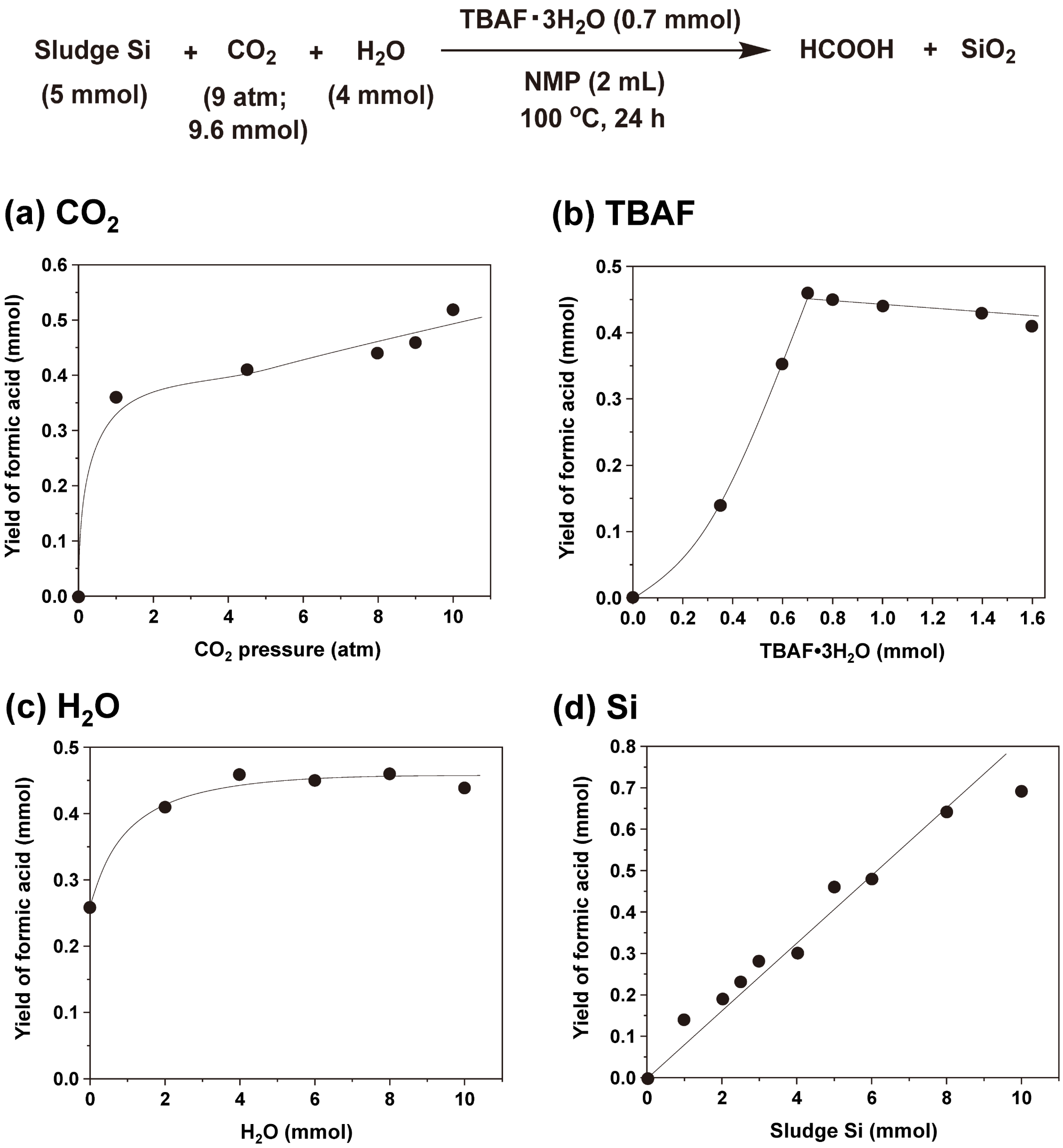
Basic conditions: Si (5.0 mmol), CO_2 (9 atm; 9.6 mmol), H_2O (4.0 mmol), TBAF-$3H_2O$ (0.7 mmol), NMP (2 mL), 100 ℃, and 24 h.

Fig. 4 (a) Time-course of the CO_2 Reduction with Sludge Si and (b) Effect of Reaction Temperature on the CO_2 Reduction

the reaction of sludge Si and CO_2. Formic acid (HCOOH) was the only carbon-derived product under the optimized conditions in liquid phase (100 ℃, 24 h). A previous report on the reaction using ^{13}C-enriched CO_2 ($^{13}CO_2$) clearly indicated that the produced HCOOH originated from CO_2[32]. Time-course of the CO_2 reduction is illustrated in **Fig. 4(a)**. The yield of formic acid increased rapidly during the initial 1-10 h and reached the maximum at 24 h (0.46 mmol). We also analyzed the gas phase after the reaction, and the formation of small amounts of CO (9.7×10^{-2} mmol) and H_2 (0.11 mmol) was detected. These values are much smaller than formic acid, indicating selective reduction of CO_2 to formic acid under the reaction conditions. Theoretically, one silicon atom produces two formic acid molecules from two CO_2 molecules. However, the formic acid amount was 0.46 mmol, and the reaction was stopped. This low productivity of formic acid is probably due to the low content of pure Si(0) in the silicon sludge. This is also supported by the high reactivity of fresh silicon powder[32]. Next, the impact of varying the reaction temperature on the production of formic acid was analyzed (**Fig. 4(b)**). The maximum formic acid yield (0.46 mmol) was attained at 100 ℃. The decrease in the amount of formic acid at higher temperatures could be due to the promotion of side reactions or a decrease in catalyst performance due to decomposition. Based on these results, subsequent optimization experiments were conducted at 100 ℃ for 24 h.

The effect of the pressure/loading amount of the substrate or catalyst on the formic acid yield is illustrated in **Fig. 5**. **Figure 5(a)** exhibits a significant increase in formic acid (HCOOH) production with the increase in CO_2 pressure, indicating that the reaction rate is affected by the CO_2 activation step. At low pressures (0-2 bar, 1 bar $= 1 \times 10^5$ Pa), the increase in the HCOOH yield was moderate, while a more substantial improvement was evident at higher pressures (8-10 bar). The CO_2 concentration could have affected the pH of the solution and increased the reaction rate; however, the details remain unclear. **Figure 5(b)** illustrates the effect of the quantity of the TBAF・$3H_2O$ catalyst on the catalytic conversion of CO_2 into formic acid. Initially, the yield increases; the highest value is attained at 0.46 mmol of formic acid with 0.7 mmol of TBAF・$3H_2O$. The amount of formic acid formed was less than that of the TBAF, however, in the case of fresh silicon, more than 0.5 mmol of formic acid was obtained with only 0.05 mmol of TBAF[32]. The reason in the lower formic acid amount with silicon sludge is thought to be (i) the lower reactivity of silicon sludge than fresh silicon powder and/or (ii) deactivation of TBAF by impurity in the sludge. The findings of our previous study indicate the catalytic role of TBAF: activation of Si-Si bond of silicon to form Si-H species[35] that react with CO_2[32]. Above 0.7 mmol, the yield of HCOOH remained almost stable.

The effect of the quantity of H_2O on the formic acid yield is illustrated in **Fig. 5(c)**. Initially, as the quanti-

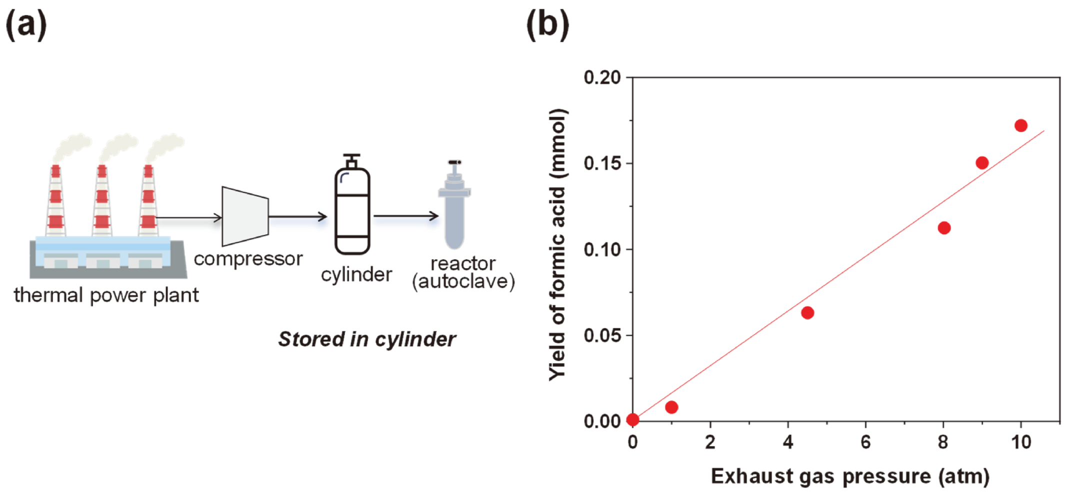


Basic conditions: Si (5.0 mmol), CO_2 (9 atm; 9.6 mmol), H_2O (4.0 mmol), TBAF-$3H_2O$ (0.7 mmol), NMP (2 mL), 100 ℃, and 24 h.

Fig. 5 Effect of (a) CO_2 Pressure, (b) TBAF Amount, (c) H_2O Amount, and (d) Si Amount on the Yield of Formic Acid in the CO_2 Reduction Reaction

ty of H_2O increased, the formic acid yield increased to 0.46 mmol, after which it became stable. Although H_2O acted as a proton source, more than 4.0 mmol of H_2O was sufficient to facilitate the protonation during CO_2 reduction. **Figure 5(d)** shows the effect of the amount of Si on formic acid production. A strong positive correlation is evident between the Si content and HCOOH yield, suggesting that saturation of formic acid yield around 0.46 mmol (**Figs. 5(a)-5(c)**) was due to the complete consumption of the reducing agent Si. No saturation point was observed within the examined range (0-10 mmol), suggesting that further increase in the Si content would increase the formic acid yield.

Figure 6(a) illustrates the method used to acquire actual exhaust gas from a thermal power plant, obtained for use in the reaction with Si. The compressed exhaust gas was stored in a gas cylinder. **Figure 6(b)** illustrates the effect of the exhaust gas pressure on formic acid production. Surprisingly, formic acid formation was observed even in the exhaust gas containing CO_2 in the presence of the Si sludge. Increasing the exhaust gas pressure positively affects the conversion to formic acid (HCOOH), suggesting that increased pressure can increase the concentration (partial pressure) of CO_2, thereby increasing the reaction rate. For the CO_2 concentration observed in the exhaust gas from the thermal power plant (14 vol%), the CO_2 partial pressure at 8 atm of total gas should have been 1.1 atm (1 atm =



Reaction conditions: Si (5.0 mmol), exhaust gas (0-10 atm), H_2O (4.0 mmol), TBAF-$3H_2O$ (0.7 mmol), NMP (2 mL), 100 ℃, and 24 h.

Fig. 6 (a) Exhaust-gas Sample Extraction from a Thermal Power Plant; Stored in a Gas Cylinder, (b) Effect of Exhaust Gas Pressure on Formic Acid Yield from CO_2 Reduction

101325 Pa). However, the amount of formic acid formed (ca. 0.12 mmol) was lower than that of 1 atm of pure CO_2 (0.35 mmol, **Fig. 5(a)**); this could have been due to the oxidation of Si in the presence of O_2 (5 vol% in the exhaust gas)[36]. The type of catalyst and/or reaction system should be further improved to enhance the reactivity of CO_2 in the exhaust gas.

3. 2. Characterization of Silicon Sludge before and after the CO_2 Reduction

Figure 7(a) illustrates the XRD patterns of the Si sludge before and after the reaction. Initially, the sample displays both Si (Si^0, PDF#75-0590) and SiC (PDF#72-0018) diffraction peaks. Silicon carbide could have formed during the silicon wafer cutting process performed using a carbon-wire saw. Following the reaction, the peaks assigned to Si^0 disappeared (suggesting a reaction between Si^0 and CO_2), while those corresponding to SiC remained. **Figure 7(b)** depicts the XPS images of the C 1s region, elucidating the changes in the carbon chemical states pre- and post-reaction. Before the reaction, the C 1s spectrum displays a peak at approximately 282.5 eV, thereby confirming the presence of silicon carbide (SiC) in the sample[37]. The SiC peak remained even after the reaction. **Figure 7(c)** presents the evolution of the chemical states of Si before and after the reaction using XPS. A strong signal at approximately 100 eV is observed before reaction. Combining the XRD and C 1s XPS analyses, this peak can be assigned to both SiC (100 eV)[37] and Si^0 (99 eV). The broad signal at approximately 102 eV might be due to SiN_x (frequently coated material on silicon surfaces). Following the reaction, the shoulder signal at approximately 99 eV (Si^0) disappeared, along with the appearance of the SiO_2 signal at approximately 103 eV. These results indicate that during the reactions, the Si^0 phase acts as a reducing agent of CO_2 conversion, while the SiC phase remains intact. **Figure 8** illustrates the image of CO_2 reduction to produce formic acid using silicon sludge containing both Si^0 and SiC phases.

4. Conclusion

This study demonstrated the potential of the sludge-derived Si as a reducing agent for CO_2 conversion to formic acid, providing a sustainable approach to repurposing industrial waste. Optimization of the reaction conditions identified the optimal fluoride catalyst dosage (0.7 mmol), CO_2 pressure (10 bar), reaction time (24 h), and water content (4 mmol) for maximizing the formic acid yield. Increasing the sludge Si quantity was found to enhance conversion efficiency directly. Characterization of the silicon sludge before and after catalysis using XPS and XRD revealed that the Si^0 phase was selectively oxidized to SiO_2 during CO_2 reduction, suggesting the role of the reducing agent Si^0, while the SiC phase remained unaltered. Although sludge Si exhibited lower reactivity compared to fresh Si[32], its effective utilization through optimization highlights its potential in scalable CO_2 valorization.

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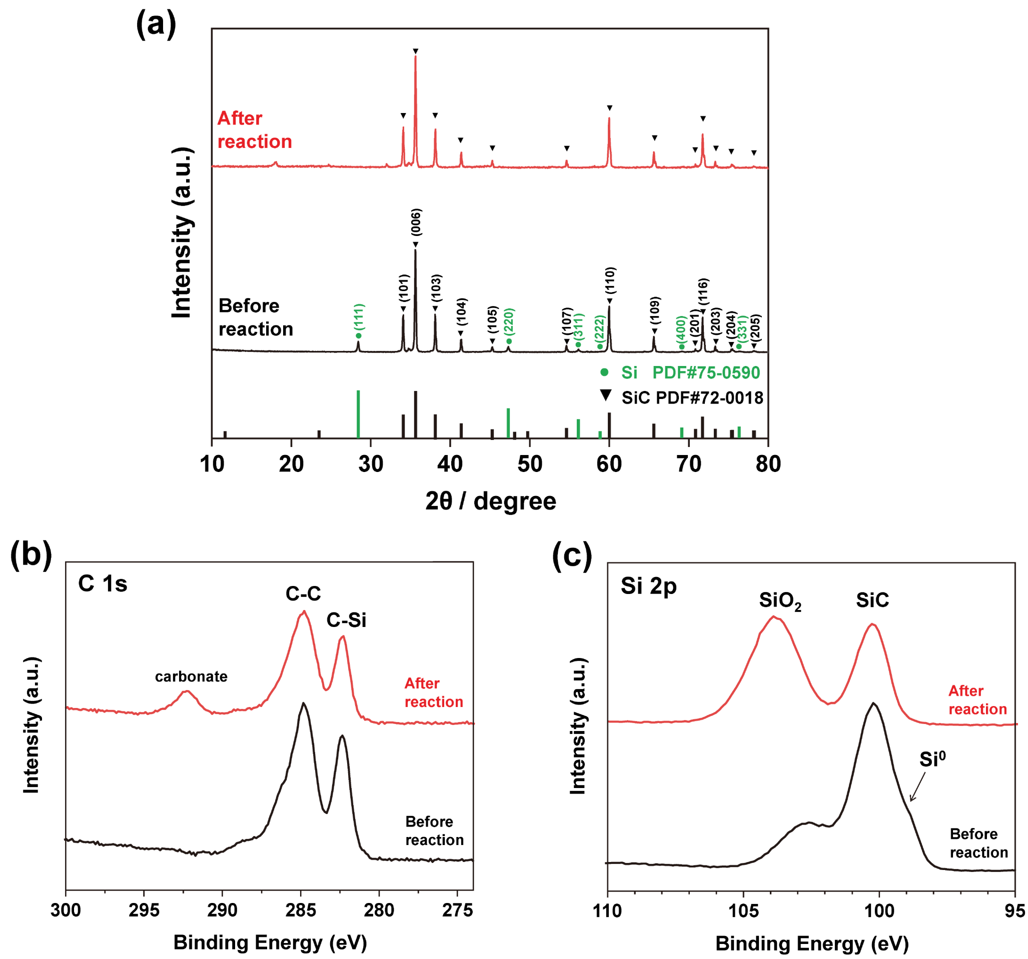

Reaction conditions: Si (5.0 mmol), CO_2 (9 atm), H_2O (4.0 mmol), TBAF-$3H_2O$ (0.7 mmol), NMP (2 mL), 100 °C, and 24 h.

Fig. 7 (a) XRD Patterns and XPS, (b) C 1s and (c) Si 2p Spectra of Si Sludge before and after CO_2 Reduction

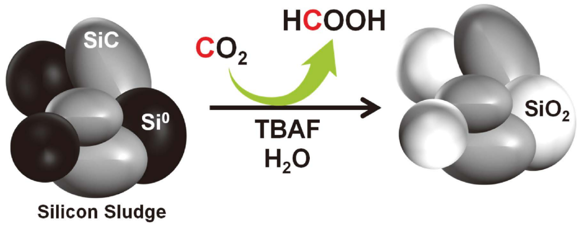

Fig. 8 Image of CO_2 Reduction to Produce Formic Acid Using Silicon Sludge

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要　　旨

シリコンスラッジを還元剤とする二酸化炭素のギ酸への変換

呉　金城[†1)]，谷村　勇亮[†1)]，劉　丹陽[†1)]，長谷川　慎吾[†1)]，
荒田　浩輔[†2)]，竹村　亮介[†2)]，難波　一夫[†2)]，本倉　健[†1)]

[†1)] 横浜国立大学大学院理工学府，240-8501 横浜市保土ヶ谷区常盤台79-5
[†2)] 電源開発(株)技術開発部若松研究所，808-0111 北九州市若松区柳崎町1番

シリコンウエハ製造工程で排出されるシリコンスラッジをCO_2から高付加価値化合物への合成に活用することで，廃棄ケイ素とCO_2の有効利用を同時に実現できる。本研究では，シリコンスラッジを還元剤とするCO_2のギ酸への変換反応を報告する。フッ化テトラブチルアンモニウムがこの反応を促進した。この手法は，純粋なCO_2だけでなく，火力発電所からの排ガス中に含まれるCO_2の変換反応にも適用可能である。CO_2圧力，反応温度，添加する水量，シリコンスラッジ量等の反応パラメーターの最適化を実施するとともに，反応前後のシリコンスラッジをXPSとXRDで解析し，シリコンスラッジ中のSi^0がSiO_2へと転化することを確認した。廃棄シリコンスラッジを活用してCO_2をギ酸へと変換する本手法は，環境調和型かつ経済的にも有利なギ酸合成法となる可能性がある。

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