Integrated Research Center for Carbon Negative Energy Science

T. Nohira, Professor
K. Kondo, Program-Specific Associate Professor

1. Introduction

To achieve carbon neutrality in 2050, it is necessary to create a new energy system which includes an active carbon dioxide fixation process in addition to "zero emission" technology. In this research center, we are investigating such carbon negative technologies. Specifically, we are engaged in research to convert carbon dioxide into useful materials using renewable energy, biomass, etc.

In this fiscal year, we have investigated the conversion of CO_2 into diamond by molten salt electrochemical process. We have also studied the activity of fungal glucuronoyl esterase to cleave ester linkages in lignin-carbohydrate complex.

2. Conversion of CO_2 into diamond by molten salt electrochemical process

As a carbon negative technology, not only the CO_2 capture and storage (CCS) but also the CO_2 capture and utilization (CCU) will play an important role in the future. Here, electrochemical CO_2 conversion technology in molten salt is considered to be one of the promising candidates for CCU [1,2]. In this study, we aimed to convert CO_2 into diamond, which is one of the most valuable carbon materials. As a first step, we investigated the electrochemical synthesis of diamond in molten LiCl–KCl systems containing K_2CO_3 and KOH, based on the premise that CO_2 dissolves as CO_3^{2-} in molten salts containing O^{2-}. We used micro-Raman spectroscopy and scanning electron microscopy (SEM) to confirm that the deposits obtained by electrolysis were diamond. As a second step, we attempted the electrochemical synthesis of diamond using CO_2 as the actual raw material. Here, we also bubbled H_2O into the molten salt to produce KOH.

Cyclic voltammetry and potentiostatic electrolysis were performed after adding K_2CO_3 and KOH to LiCl–KCl eutectic melts under an Ar atmosphere at 973 K. The working electrode was a Ni flag (Φ 3 × 0.1 mm) or Ni plate (5 mm × 10 mm × 0.1 mm), the counter electrode was a glass-like carbon rod, and the reference electrode was an Ag^+/Ag electrode. The potential was calibrated with Li^+/Li potential. After electrochemical measurements were performed, samples were prepared by potentiostatic electrolysis using the Ni plate electrodes. In the case where CO_2 was used as a raw material, LiCl–KCl eutectic molten salt containing Li_2O was prepared, and CO_2 and H_2O were bubbled into it in predetermined amounts, respectively. After electrochemical measurements, potentiostatic electrolysis was performed to prepare samples. The obtained samples were analyzed by SEM, EDX, and micro-Raman spectroscopy.

First, electrochemical measurements in baths containing only K_2CO_3, only KOH, or both indicated that carbon deposition and hydrogen evolution proceed simultaneously in the potential range more negative than 1.2 V. Then, samples were prepared by potentiostatic electrolysis using Ni plate electrodes at 1.0 to 1.2 V (vs. Li^+/Li). As an example, the surface SEM image of a deposit obtained at 1.1 V is shown in Fig. 1a. The EDX analysis of this area showed that only C and Ni from the substrate were detected. The result of micro-Raman spectroscopy of the sample is shown in Fig. 1b, which shows a spectrum characteristic of diamond with a sharp peak at 1332 cm^{-1}. Based on the results of these three analyses, the electrochemically synthesized angular particles were identified as diamond.

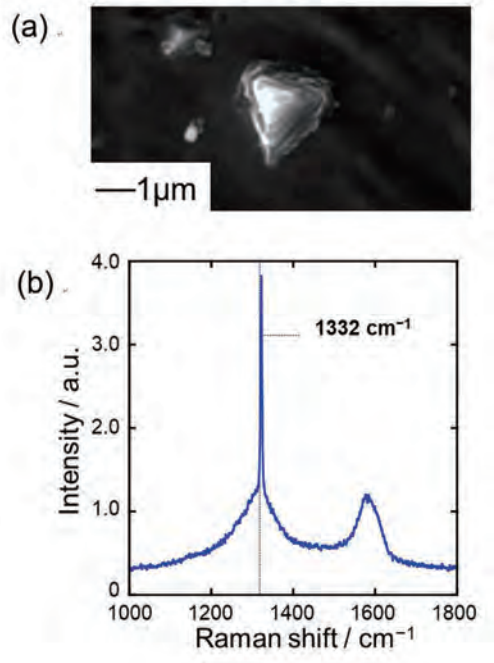

Fig. 1 (a) An SEM image and (b) a Raman spectrum of the sample obtained by potentiostatic electrolysis of a Ni plate electrode in molten LiCl–KCl–K_2CO_3–KOH at 973 K.

Second, CO_2 gas and H_2O gas were introduced in molten LiCl–KCl containing O^{2-} ions. After that, potentiostatic electrolysis was performed at 1.0 to 1.2 V (vs. Li^+/Li). In samples prepared at 1.2 V, we observed a peak at 1332 cm^{-1} attributed to diamond, as well as broad peaks attributed to the D-band and G-band of amorphous carbon. The obtained deposits are considered to be a mixture of diamond and amorphous carbon.

3. Activity of fungal glucuronoyl esterase to cleave ester linkages in lignin-carbohydrate complex

A major component of woody biomass is a lignin-carbohydrate complex which is formed by associations of lignin, hemicellulose, and cellulose. Lignin is an attractive aromatic resource for the production of various materials. Hemicellulose and cellulose can also be utilized to produce biofuels and multiple materials. However, the recalcitrant structure of the lignin-carbohydrate complex is an obstacle to the efficient utilization of woody biomass. In the lignin-carbohydrate complex, the association of lignin and hemicellulose is strengthened by covalent cross-linkages such as ether, ester, and phenyl glycoside linkages. Among these linkages, ester linkages formed between lignin and glucuronoxylan are often found in hardwood biomass, where glucuronoxylan is a main hemicellulose component. Glucuronoyl esterase (GE) is an enzyme that catalyzes the hydrolysis of the ester linkage, thereby weakening the association of lignin and glucuronoxylan (Fig. 2).

We previously identified a fungal GE which exerts high catalytic efficiency toward a model substrate, benzyl glucuronate. Although the GE can be prepared by yeast expression system, its yield (0.16 mg/L-culture) was insufficient for the investigation of GE activity toward natural substrates. This year, we improved the purification procedure for the GE. As a result, the yield of the GE reached 3.65 mg/L-culture. Therefore, a 28-fold increase in the yield was achieved.

We then investigated the activity of the GE toward ester linkage in the natural woody biomass. Firstly, a biomass fraction rich in the ester linkage was extracted from beech wood powder. From the extracted fraction, multiple signals of ester linkage were observed by NMR spectroscopy, which suggested the existence of various structures of lignin or hemicellulose proximal to the ester linkage (Fig. 3a). Next, enzymatic reaction by the GE toward the extracted fraction was performed. Intensities of some ester linkage signals were reduced by the reaction (Fig. 3-2b, labeled with asterisks), which indicated successful cleavage of ester linkages catalyzed by the GE. Exceptionally, the intensity of one ester linkage signal was not changed by the reaction (Fig. 3b, labeled with an arrow head). This result suggested that GE could not cleave the ester linkage giving this signal. The proximal structure of lignin or hemicellulose to this ester linkage may prevent GE activity due to steric hindrance. Our NMR analysis demonstrated the GE activity to cleave ester linkages in the lignin-carbohydrate complex extracted from natural beech wood. In addition, an insight into the preference of substrate structure for GE activity was obtained.

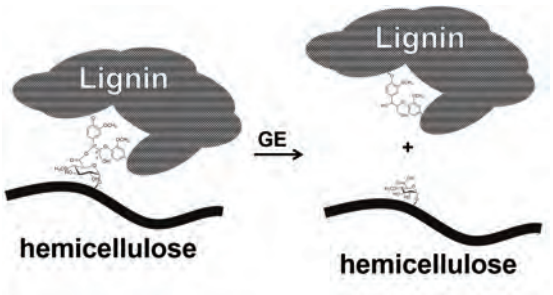

Fig. 2. Cleavage of ester linkage between lignin and hemicellulose.

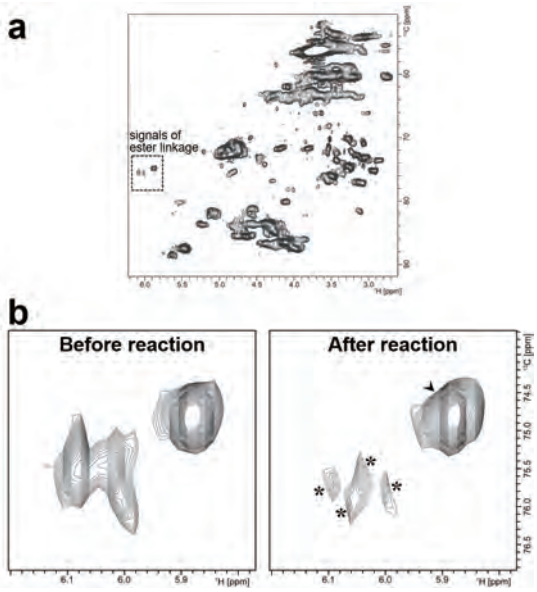

Fig. 3. NMR spectrum of extracted lignin-carbohydrate complex fraction (a) and ester linkage signals before and after the reaction by the GE (b).

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