MERIT Internship Report

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Yamaguchi Laboratory, Department of Applied Chemistry, School of Engineering
D3 / MERIT-WINGS 9th student
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Overview

Hosting company	Interdisciplinary Research Center for Catalytic Chemistry, AIST
Period of stay	July 3rd, 2023 ～ July 31st, 2023 (1 month)
Research title	Synthesis of redox raw materials utilizing flow reaction system

※ Since the details of the research are confidential, this report refers only to an overview.

Background

A redox flow battery is one of the very promising technologies in terms of energy storage because of its excellent durability, response, discharge ability, and so on. In addition, the energy capacity can be scaled up easily by increasing the volume of the storage tank. However, compared with the conventional batteries where the energy is stored in solid electrodes, the energy density of the redox flow battery composed of aqueous electrolytes is lower, and the increase of both cell potential and the concentration of the redox-active species is required. Therefore, nonaqueous redox flow batteries, in which redox-active organic molecules are dissolved in a nonaqueous electrolyte with a wider electrochemical stability window than water, are attracting attention. Although reduction of the precursor and the subsequent functionalization are needed to synthesize redox-active organic molecules, or redox raw materials, isolation of the intermediates is very difficult owing to their instability. Applicable compounds that can be synthesized through a one-pot procedure are still quite limited.

A liquid-phase continuous-flow reaction system using a heterogeneous catalyst can provide high activity and selectivity without formation of by-products for a long period by appropriately selecting catalyst packed columns and controlling the flow rate of the substrates. It does not need separation from the catalyst and allows the synthesis and functionalization of unstable intermediates with high purity. Thus, this reaction system is an excellent method for highly efficient synthesis of not only redox raw materials but also various organic molecules.

During this period, I developed catalytic synthesis of organic molecules useful as redox raw materials by a continuous-flow reaction system. Since I have been majored in inorganic chemistry recently and will work as a researcher in a company after graduation, I expect to expand my

experimental techniques by carrying out research in a new environment with an industrial perspective, which is different from my own research, and to make use of this experience from the next year onward.

Activity contents

- Synthesis, isolation, and characterization of the substrate for the continuous-flow reaction
- Learning a series of operations related to the liquid-phase flow reaction system
  (from preparation of catalyst packed columns to analysis of reaction results)
- Reaction screening using several kinds of catalysts

Impressions

I experienced the use of a continuous-flow reaction system for the first time and learned a lot. The system itself was simpler than I had expected, but it was reasonably designed and easy to operate, and the advantages described in [Background] were very important. This time I used the small-scale columns for catalyst screening, but there were also columns for ultra-scale (kg) that were used by companies to monitor practical applications, and I was able to watch them. Even though it was different from my own field, as I can study only on a laboratory scale, it was a valuable opportunity for me to visualize the process leading up to practical application more closely. In this screening, I was very glad to obtain good findings indicating a clear difference in activity depending on the catalysts.

The experience of experimenting in a different environment was valuable and stimulating. Unlike our laboratory full of students, the research team consisted of people from various backgrounds was very calm. The most unfamiliar difference for me was that the number and arrangement of the draft chambers were different and the place where we could handle organic solvents was limited. Anyone will experience such changes in the experimental environment many times in the future, and it is very meaningful for me to be able to experience them while I am still a student. I would like to recommend such experience not only to MERIT students but to other students as well. I was also able to hear from a person who had worked for a company for a long time about his experiences at that time, which was helpful for me.

Acknowledgements

I would like to express my gratitude to AIST Interdisciplinary Research Center for Catalytic Chemistry Innovative Oxidation Team Leader Dr. Kon and Dr. Miyamura for accepting me for this internship. I would like to thank the members of the team for teaching me about the operation and the rules of the laboratory and the team. I would also like to thank my supervisor Prof. Yamaguchi and secondary supervisor Prof. Takanabe for the support and the agreement of this internship, and MERIT program for providing me this valuable opportunity.