Electrosynthesis using New Biphasic Organic Reaction System with Mutual Miscibility

Kazuhiro Chiba, Yusuke Kono, Shokaku Kim, Yoshikazu Kitano, Masahiro Tada

Laboratory of Bio-organic Chemistry, Tokyo University of Agriculture and Technology 3-5-8 Swaiwai-cho, Fuchu, Tokyo 183-8509, Japan

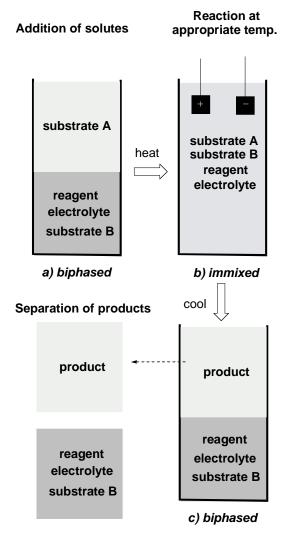
Combinatorial chemistry has developed into an important means of drug discovery. However, to fully empower this technology, it remains necessary to adapt the construction of more complicated reactions and molecules to the solid-phase, liquid-phase, or fluorous system methods. As more and more fluorous reagents become available, the possibilities for liquid-phase combinatorial synthesis in a spatially separated mode will expand. In the field of electrosynthesis, liquid-liquid separation system should play important roles that enable the products-electrolytes or products-reagents separation after electrolysis. Our group thereby has much interest in the expectative novel liquid-liquid separation systems for electrochemical applications.

Recently, we investigate some electrolytic intermolecular reactions in lithium perchlorate / nitromethanes.¹⁻⁴⁾ The electrolyte solution showed marked property in the acceleration of the intermolecular carbon-carbon bond formations and in the stabilization of intermediates. The media effect gave us the incentive to try to determine whether such electrochemical media could be applied for the construction of new, electrolytic high-throughput and eco-friendly reaction system.

In this paper, we describe our initial finding of the liquid-liquid reaction systems using typical organic solvents that are perfectly miscible by a moderate, thermo-control in arbitrary ratios of upper and lower layers. The reaction system was simply constructed by mixtures of some qualified less-polar and polar organic solvents, enabling the introduction of a wide variety of chemicals. By using this system, designed less-polar solutes and polar solutes in a one-phase solution were spatially separated by cooling the reaction mixture with the exclusion of the less-polar phase onto the polar, lower-phase solvents. This property of our system could be an innovative tool for general organic reactions and liquid-phase combinatorial chemistry with many advantages of electrochemical applications, ease of compound separation, and handling.

References

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Miscible between two separated liquid phases of different polarity ?

Miscible at an arbitrary ratio of upper and lower phases ?