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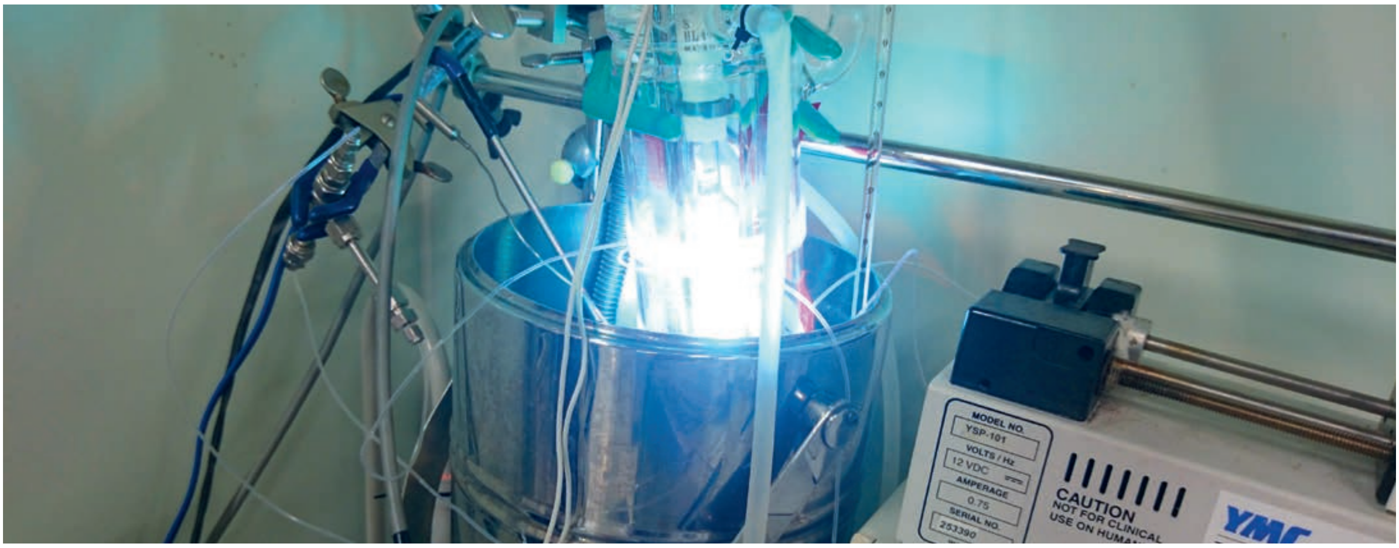

The photoreactor used to produce cyclobutanes.

Photochemistry

Microflow set-up boosts output

Photochemical reactions in a flow rather than a batch reactor lead to easy scaling up

Most chemical reactions require energy to start them. In almost all cases, that energy is heat, but for certain reactions, light energy must be used instead. Known as photochemical reactions, these include one of the most effective ways of producing cyclobutane rings — cyclic structures containing four carbon atoms, which are frequently found in naturally occurring compounds with useful biological properties, such as insect pheromones.

Now, Yasuhiro Nishiyama and his team at NAIST's Graduate School of Materials Science in Japan, along with Michael Oelgemöller at Australia's James Cook University, have shown that photochemical reactions like these can be done very effectively in a flow chemistry set-up[1].

Most industrial chemical processes are run in a so-called 'batch set-up', which is not unlike the procedure for baking a cake: reactants are combined in known proportions and mixed for a set time. Each reaction batch produces a fixed amount of product. But this way is not very amenable to photochemical reactions: "Light cannot penetrate sufficiently into large batch reactors or high-concentration solutions," explains Nishiyama. "Thus, almost all photoreactions show poor conversion in batch reactors."

> **"If we need to scale up, we can also run multiple reactors in parallel, a process called numbering up."**

The team investigated reactions that produce cyclobutanes from two alkenes (a class of hydrocarbons containing only carbon and hydrogen) in a microflow set-up. In this set-up, the reaction tubes have an internal diameter of just 1 millimetre and are wrapped around the light source, allowing the whole apparatus to be cooled easily (see figure).

Of particular interest is that one of the reagents — ethylene — is a gas. In a batch process, this would add a further difficulty, since the reaction can only occur at the surface between the gas and liquid reagents. In flow, however, pumping a gaseous and a solution reagent into a small tube produces small gas bubbles in the solution as it flows through the tube. This is known as slug flow, and it ensures that there is a very thin film of reactant solution at the edge of the tube, which is easily penetrated by light. The small bubbles create a large surface area at which the reaction can occur.

Photoreactions performed in the flow set-up are slightly more efficient and selective than those performed in a small batch reaction. Importantly, making a large amount of product is much easier: simply running the reactor continuously produces more product. "If we need to scale up, we can also run multiple reactors in parallel, a process called numbering up," says Nishiyama.

Reference

1. Terao, K., Nishiyama, Y., Tanimoto, H., Morimoto, T., Oelgemöller, M. & Kakiuchi, K. Diastereoselective [2+2] photocycloaddition of a chiral cyclohexenone with ethylene in a continuous flow microcapillary reactor. *Journal of Flow Chemistry* **2**, 73–76 (2012).

More information about the group's research can be found at the Synthetic Organic Chemistry Laboratory webpage:
http://mswebs.naist.jp/english/courses/1414/