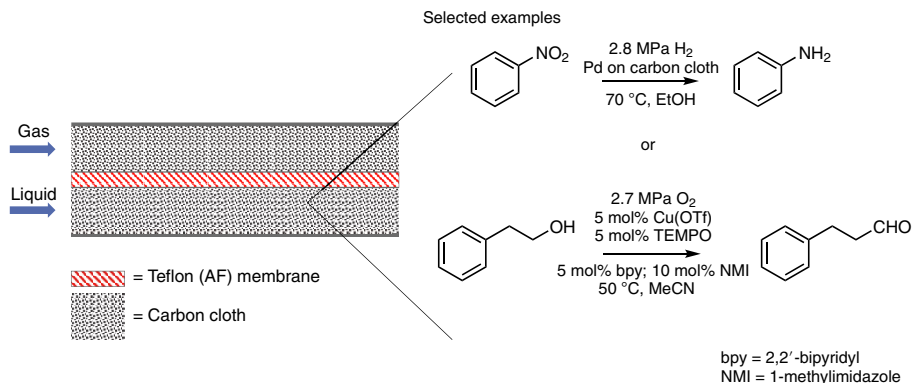


MICROREACTOR CATALYSIS

Flowing through stacks

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Credit: Davide Esposito

The repertoire of pharmaceutical synthesis encompasses a variety of multiphase catalytic reactions, which include gas–liquid hydrogenation or oxidation of key intermediates. Such processes, however, involve handling high-pressure flammable gas–liquid mixtures and very often raise practicality and safety concerns. Now, Klavs Jensen and co-workers at the Massachusetts Institute of Technology report a thin-layer membrane microreactor design that allows for both heterogeneous and homogeneous catalytic gas–liquid reactions to be performed safely and efficiently.

The reactor is based on a teflon amorphous fluoroplastic (AF) membrane stacked between two sheets of thin-layer carbon cloth (pictured). The porous carbon layers transport either the gas or the liquid feeds, and can be used as support for heterogeneous catalysts. Additionally, the membrane provides the system with excellent gas–liquid mass transfer performance by minimizing the diffusion distance between the gaseous reagents and the catalyst. The authors tested the practicality of the system by performing the high-pressure palladium-catalysed hydrogenation of nitro- or carbon–carbon-multiple-bond-containing substrates. Excellent preparative yields could be obtained with very short residence times of 0.5 to 1 minute. Experiments on

the homogeneous Cu/TEMPO-catalysed oxidation of different alcohols in the presence of oxygen additionally showed the versatility of the system.

Reducing the size of the carbon cloth layer favours mass transfer limited reactions, whereas kinetically limited reactions can be enhanced by using thicker carbon layers, as shown by modelling studies aimed at the optimization of the reactor design. This approach offers the possibility of balancing the productivity of the process with the reactor manufacturing costs. Moreover, the construction facilitates process scale-up by simply stacking multiple membrane reactor layers, which operate in parallel under a uniform flow regime. As an example, a three-layer membrane reactor proved competent to perform the catalytic hydrogenation of ethyl cinnamate with a productivity of 1.9 g h^{-1} , threefold higher than in the case of a single layered reactor. Such microreactor designs are expected to facilitate the application of multiphase catalytic reactions in pharmaceutical and synthesis research laboratories.

Davide Esposito

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