

Flow Chemistry Highlights

A CHIMIA Column

Review of Recent Literature on Flow Chemistry. Selected Topic: Versatility

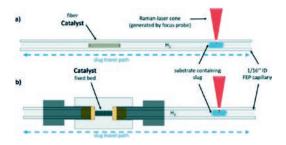
Oscillating droplet reactor – towards kinetic investigations in heterogeneous catalysis on a droplet scale

Torsten Klement, Schirin Hanf, Fabian Wolff, Norbert Kochmann, Stephan A. Schunk, Thorsten Röder*, *React. Chem. Eng.* **2021**, *6*, 1023, https://doi.org/10.1039/D0RE00466A

While heterogeneous catalysis is a highly impactful area in chemistry, it is notoriously challenging to scale-up. The multiphase nature requires that kinetics are determined under realistic reaction conditions, which can include gases under elevated pressures and non-ideal heat-flows. The authors chose to study hydrogenations catalyzed by palladium on carbon supports, and established a packed-bed reactor within capillary tubing. The chemical kinetics of these three-phase systems could be determined by contactless in-line Raman spectroscopy, with the probe registering the periodic passage of an oscillatory slug of reaction mixture passing back and forth along the tubing. In this way, the reaction time is decoupled from the flow-rate or reactor length, and kinetic plots from the nitro group and olefin reductions could be obtained from only small amounts of materials. The kinetic effects of hydrogen pressure-variation and mean catalyst particle size could be evaluated, and the setup proved itself also for slower reactions. With this foundation the authors foresee usage with a wide variety of solid catalysts, gases and substrates.

Author's comments*:

"We see this approach as a universal screening machine for process development and determination of the kinetics. We have already used these successfully for the investigation of polymerization. Another high-potential application is catalyst screening in homogeneous catalysis."



Design and Characterization of a Scaled-up Ultrasonic Flow Reactor

Claire Delacour, Dwayne S. Stephens, Cécile Lutz, Robert Mettin, Simon Kuhn*, *Org. Process Res. Dev.* **2020**, *24*, 2085, https://doi.org/10.1021/acs.oprd.0c00148

The handling of solids is one of the major issues in flow chemistry: particulate material accumulates and blocks fluidic connections, causing loss of material and operation time and requiring tedious clean-up. Ultrasonic reactors, in which acoustically induced bubble cavitation breaks up particle agglomerates, promise to alleviate this problem, albeit that their application at scale is rare. In this study, the authors successfully demonstrate how an ultrasonic reactor may be scaled-up. First, they validate CFD simulations with experimental measurements to map the acoustic pressure distribution within the reactor. Subsequently, they investigate yield and particle size distribution in barium sulfate formation at various configurations and frequencies of the acoustic transducers to identify conditions with high and stable yield and lowest clogging likelihood. CFD computations link observed improvements with pressure fields and thus cavitation activity; such an approach could be followed in the future to further scale the process or design novel reactors. The final scale-up was performed reliably for about 2 hours at a production scale of 14 g/h. While this remains modest for many industrial applications, the study shows a promising way forward for solids handling in flow systems at scale.

Author's comments*:

"This work highlights how the combination of theoretical and experimental characterization supports the design of novel ultrasound reactors, further promoting process intensification and sustainable manufacturing."

