Micro flow synthesis enabled by a supported aqueous phase catalyst coating

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Abstract title	MICRO FLOW SYNTHESIS ENABLED BY A SUPPORTED AQUEOUS PHASE CATALYST COATING
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Micro flow processing provides many advantages, such as improved heat- and mass transfer and the potential for seamless integration of process steps.[1] However, as with all continuous flow processes, the application of organometallic catalysts is still hampered by the need for catalyst recovery.[2,3] Although methods for catalyst retention can be employed as an alternative, these typically suffer from issues such as high cost, reduced performance and catalyst deactivation.[3]

The use of a micro flow system provides a distinct advantage; process control. By “fluids structuring”, the organometallic catalyst can be made heterogeneous by containing it within a fluid captured in the microreactor, thus preserving its homogeneous properties and performance. In addition, the non-permanent nature of this retention method allows a straightforward recovery and recycling of the catalyst in case of deactivation. This presentation will show the results of our exploration of innovative reaction environments within the field of Novel Process Windows.[1]

To investigate the application of a Supported Aqueous Phase Catalyst (SAPC) coating for the Mizoroki-Heck reaction, an organometallic palladium SAPC was prepared based from both silica powder and from a silica coated capillary. These were then applied as catalysts in batch (SAPC powder) and in micro flow (SAPC coating) for the Mizoroki Heck reaction, see Figure 1.[4]

In micro flow, application of the SAPC coating for the Mizoroki-Heck reaction provided a 21% yield after 2.9 min, using only 0.01 mol% catalyst loading. This compared favorably to batch experiments, which provided a 83% product yield after 4 h using 0,4 mol% catalyst loading. In addition, salt accumulation in batch prevented re-use of the catalyst after 3 runs, an issue that could be overcome in micro flow by the continuous removal of these salts.

These results demonstrate the advantage of applying the SAPC as a coating in micro flow. Further investigation of the application of fluids structuring will be focused on the hydroformylation of octene in supercritical carbon dioxide at pressures up to 300 bar, using an organometallic rhodium catalyst. Tunable fluids such as supercritical carbon dioxide have properties that can be adjusted by regulating temperature and pressure, providing further means for process control and optimization.

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[3] Hintermair, U., Giancarlo, F., Leitner, W., Chem. Commun. 47 (2011) 3691.

[4] Stouten, S., Wang, Q., Noël, T., Hessel, V., manuscript in preparation

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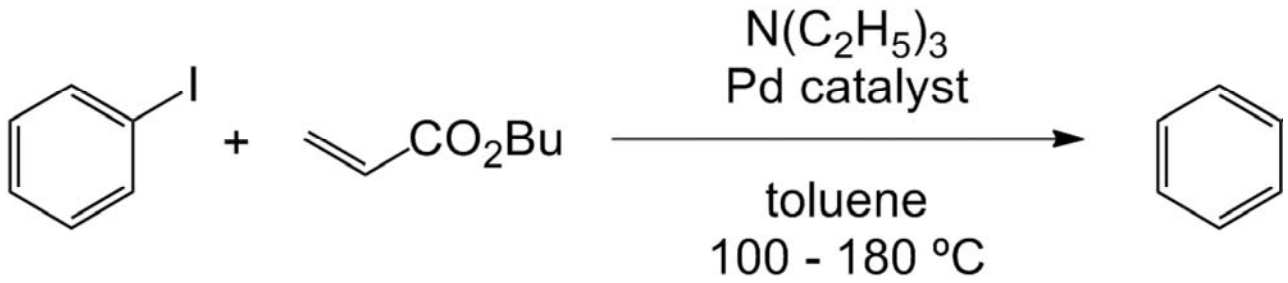
www:http://w3.chem.tue.nl/en/the_department/research_groups/micro_flow_chemistry_process_technology/

Images



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