Continuous sol-gel synthesis of phosphated TiO_2 catalysts in a microreactor

O. Martin[1], N. Bolzli[1], P. Riedlberger[1]*

[1]Institute of Chemistry and Biotechnology, Zurich University of Applied Sciences

Continuous microreactors, featuring µm-sized tubes and thus a fast mass and heat transfer, enable a precise control of chemical processes, which leads to improved efficiency and safety with respect to conventional large-scale reactors.[1] In addition, their modular design adds unrivaled flexibility. While numerous catalyzed reactions in microreactors have been reported,[1] the preparation of heterogeneous catalysts in these systems has been greatly overlooked even though a controlled design of catalysts will be key for the development of more sustainable chemical processes.[2]

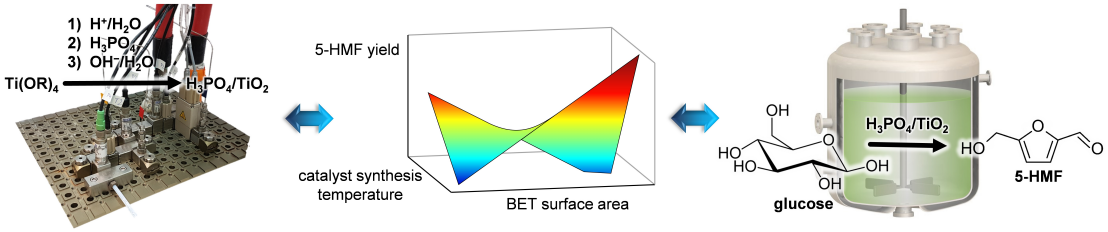

Figure 1. The DoE strategy applied herein correlates (center) the parameters of the preparation of phosphated TiO_2 *via* a novel continuous sol-gel route in a microreactor (left) with the resulting catalyst properties as well as the catalytic performance upon the 5-HMF synthesis from glucose (carried out in a batch reactor).

Therefore, we established a novel continuous sol-gel microreactor process for the production of phosphated TiO_2, which can be applied as cheap and eco-friendly catalyst for the selective synthesis of the platform chemical 5-hydroxymethylfurfural (5-HMF) from bio-derived glucose.[3] This approach allows a highly efficient catalyst preparation since all steps, *i.e.*, (*i*) sol formation, (*ii*) functionalization by H_3PO_4, and (*iii*) gel formation, are carried out in one continuous process (**Figure 1**, left). The catalytic performance was correlated with the catalyst properties (*e.g.*, surface area) and its synthesis parameters (*e.g.*, temperature) through design of experiments (DoE, **Figure 1**) to obtain the optimal catalyst preparation process and the material with the best catalytic results. Due to these correlations, we could additionally gather a deeper mechanistic understanding of both catalyst synthesis and catalyzed reaction. This was essential to rationalize phenomena occurring such as fouling, *i.e.*, blockage of the microchannels by large agglomerates, which can pose a critical challenge upon the catalyst production.

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[3] L. Atanda, S. Mukundan, A. Shrotri, Q. Ma, J. Beltramini, *ChemCatChem,* **2015**, *7*, 781–790.