

Continuous sol-gel synthesis of phosphated TiO₂ catalysts in a microreactor

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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]

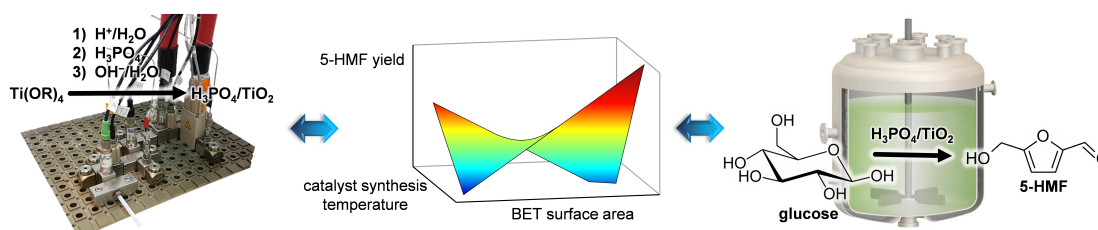


Figure 1. The DoE strategy applied herein correlates (center) the parameters of the preparation of phosphated TiO₂ via a novel continuous sol-gel route in a microreactor (left) with the resulting catalyst properties as well as the catalytic performance upon the 5-HMF synthesis from glucose (carried out in a batch reactor).

Therefore, we established a novel continuous sol-gel microreactor process for the production of phosphated TiO₂, 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 H₃PO₄, 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.

[1] V. Hessel, S. Hardt, H. Löwe, *Chem. Micro Process Engineering*, Wiley-VCH, Weinheim, **2004**, 674 pp.

[2] H. Jacobsen, in *Handbook of Green Chemistry, Vol. 2* (Eds.: T. Anastas, R. H. Crabtree), Wiley-VCH, Weinheim, **2013**, pp. 93–116.

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