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Sonication as a Versatile Tool for Photocatalysts' Synthesis and Intensification of Flow Photocatalytic Processes Within the Lignocellulose Valorization Concept

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Abstract : This work is a report of recent selected experiments of photocatalysis intensification using flow microphotoreactors (fabricated by an ultrasound-based technique) for photocatalytic selective oxidation of benzyl alcohol (BnOH) to benzaldehyde (PhCHO) (in the frame of the concept of lignin valorization), and the proof of concept of intensifying a flow selective photocatalytic oxidation process by acoustic cavitation. The synthesized photocatalysts were characterized by using different techniques such as UV-Vis diffuse reflectance spectroscopy, X-ray diffraction, nitrogen sorption, thermal gravimetric analysis, and transmission electron microscopy. More specifically, the work will be on: a Design and development of metal-containing TiO_2 coated microflow reactor for photocatalytic partial oxidation of benzyl alcohol: The current work introduces an efficient ultrasound-based metal (Fe, Cu, Co)-containing TiO_2 deposition on the inner walls of a perfluoroalkoxy alkanes (PFA) microtube under mild conditions. The experiments were carried out using commercial TiO_2 and sol-gel synthesized TiO_2. The rough surface formed during sonication is the site for the deposition of these nanoparticles in the inner walls of the microtube. The photocatalytic activities of these semiconductor coated fluoropolymer based microreactors were evaluated for the selective oxidation of BnOH to PhCHO in the liquid flow phase. The analysis of the results showed that various features/parameters are crucial, and by tuning them, it is feasible to improve the conversion of benzyl alcohol and benzaldehyde selectivity. Among all the metal-containing TiO_2 samples, the 0.5 at% Fe/TiO_2 (both, iron and titanium, as cheap, safe, and abundant metals) photocatalyst exhibited the highest BnOH conversion under visible light (515 nm) in a microflow system. This could be explained by the higher crystallite size, high porosity, and flake-like morphology. b. Designing/fabricating photocatalysts by a sonochemical approach and testing them in the appropriate flow sonophotoreactor towards sustainable selective oxidation of key organic model compounds of lignin: Ultrasonication (US)-assitedprecipitaion and US-assitedhydrosolvothermal methods were used for the synthesis of metal-oxide-based and metal-free-carbon-based photocatalysts, respectively. Additionally, we report selected experiments of intensification of a flow photocatalytic selective oxidation through the use of ultrasonic waves. The effort of our research is focused on the utilization of flow sonophotocatalysis for the selective transformation of lignin-based model molecules by nanostructured metal oxides (e.g., TiO_2), and metal-free carbocatalysts. A plethora of parameters that affects the acoustic cavitation phenomena, and as a result the potential of sonication were investigated (e.g. ultrasound frequency and power). Various important photocatalytic parameters such as the wavelength and intensity of the irradiated light, photocatalyst loading, type of solvent, mixture of solvents, and solution pH were also optimized.

Keywords : heterogeneous photo-catalysis, metal-free carbonaceous materials, selective redox flow sonophotocatalysis, titanium dioxide
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