# Structural and morphological characterization of VPO catalysts

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Vanadium phosphorus oxides, (VPO) are commercially used as catalysts for the oxidation of *n*-butane to maleic anhydride (MA). A number of methods have been proposed for the catalyst preparation and considerable efforts have been applied in order to get an understanding of the nature of the active site and to improve the catalytic performance [1]. The conversion and selectivity rate of a VPO catalyst is highly sensitive to the phase composition and to the morphology and microstructure, which in turn are determined by preparation and activation processes.

In this work we present a comprehensive characterization of VPOs prepared by reduction of $V_2O_5$ and $VO_2$ respectively. A combination of methods are applied: scanning electron microscopy (SEM) for the information of morphology, energy dispersive x-ray analysis (EDX) for qualitative and quantitative elemental analysis (both on a Hitachi S-4000 SEM); X-ray diffraction (XRD) for phase analysis (on a Stoe Powder Diffractometer with the position sensitive detector); TEM for microstructural analysis – operating in low magnification, high resolution images mode and diffraction patterns – (on Philips CM 200 FEG electron microscope). Samples suitable for TEM investigations were prepared by dispersing the catalyst powder into a carbon film supported on a copper mesh grid.

Four catalysts were investigated: SAP 1, prepared by reduction of $V_2O_5$ in a $H_3PO_3$, $H_2O$, $H_3PO_4$ solution; SAP 2, prepared as SAP 1 but followed by reflux in $H_2O$; SAP 3, prepared by reduction of $V_2O_4$ in a $H_2O$, $H_3PO_4$ $H_4P_2O_7$ solution; and SAP 4, prepared as SAP 3 but followed by reflux in $H_2O$.

The results from SEM show that the surface morphology of the investigated samples are different, ranging from thin and plate-like crystals (SAP 1), to well-defined cubic crystals with almost the same distribution within the limited range of investigation (SAP 3). Sample SAP 2 seems to have larger surface area and this can be the underlying reason for the best conversion rate (65%) among the studied four samples. However, quantitative elemental analysis exhibits very similar chemical constitutions for all catalysts. Study of the x-ray powder diffraction patterns show the presence of the main $(VO)_2P_2O_7$ (tetravalent phase) and other $V^{5+}$ phases. TEM studies show that these powders are very sensitive to the electron beam and amorphous overlayers can occur on the edge of the platelets. The best catalyst exhibits the most defective internal structure and the smallest crystallite size. More detailed results from XRD and TEM including image matching will be presented.

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Reference:
1. for instance, the papers in the special issue of Catalysis Today 16 (1993)