



COMPARSION OF TRIBOMECHANICAL AND CHEMICAL ACTIVATION OF VPO

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The reactivity of solids depends on their surface properties. Generally the properties vary with the conditions of the synthesis and post- treatment, which determine the texture, particle size, specific surface area, type of crystal faces exposed and the number of active sites [1 & 2]. Mechanical treatment of VPO catalyst causes a substantial increase of both catalytic activity in *n*-butane oxidation and the selectivity to maleic anhydride. Changes in specific surface area and anisotropic deformation take place. Mechanochemical treatment of the initial reagent impairs such properties that they remarkably influence the catalytic properties of the final catalyst.

VOHPO₄·0.5H₂O was mechanotreated in ethanol and air for a duration of time and the morphology studied under SEM, TEM and by XRD. This was compared with the morphology of the initial reagent and sample thermally treated in a vacuum. Also the product $(VO)_2P_2O_7$, which was obtained from $VOHPO_4$ ·0.5H₂O was compared.

The mechanochemical treatment firstly causes repeated fractures of the solid, diminishing its particle size and corresponding increase of the specific surface area. SEM shows that the initial sample mainly of flat smooth needle like particles, which are held together like a blossom. Under mechanochemical treatment the blossom morphology is lost and layer formation forms, with more circular particles. Under hard mechanical load it takes only a short time to reach an ultimate size of the particles. XRD shows that milling in ethanol for 5 minutes broadened the peaks, indicating smaller particle size but did not change the phase. Milling in air for 28 minutes gave an amorphous phase also supported by the TEM diffraction pattern which shows a diffused ring pattern. The thermally activated sample shows a phase change to $(VO)_2P_2O_7$ but minor phase of the initial material is still present.

Chemically activated catalysts show the morphology from SEM analysis ranges from thin and plate-like to well-defined cubic crystals. Quantitative analysis exhibits very similar chemical constitutions for all catalysts. Analysis of the diffraction patterns prove, that we have a mixture of V^{5+} phases among the main tetravalent phase $-(VO)_2P_2O_7$. The XRD patterns from the chemically activated catalyst show multiphasic materials. The main phase being $(VO)_2P_2O_7$, with minor phases of α -, β -, γ -, and δ - $VOPO_4$.

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