Characterisation of tribochemically activated vanadium oxide V_2O_5

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Tribochemical treatment can be used to activate catalysts. For instance, ball-milling can modify surface/defect structure and electronic properties of catalytic materials, can reduce particle size and increase the specific surface area [1]. Improvements in the catalytic performance are reported [2]. In the present work, we study the tribochemically activated V_2O_5 by means of scanning and transmission electron microscopy (SEM and TEM), X-ray and electron diffraction, high-resolution imaging and electron energy-loss spectroscopy (EELS). Ball-milling was carried out in a planetary ball-mill: 60 g of V_2O_5 together with six agate balls (1.5 cm diameter, 11g) were placed into an agate vessel (250 cm^3 volume). Milling was performed for up to 20 h at approximately 150 revolutions per minute. Samples studied in the present work were taken after 3, 5, 10 and 20 h.

SEM and TEM-micrographs reveal the morphological development of V_2O_5 at various periods of milling in the planetary ball-mill. Up to 3 h the main effect is the crushing of large crystallites. Further milling up to 10 h leads to the formation of fine particles whose size distribution becomes narrow and samples appear more homogenous in size. The increasing of milling time up to 20 h leads to reagglomeration of small particles. X-ray powder patterns from the four milled samples show significant changes in the integral intensities of individual Bragg peaks, in the full width at half maximum and in the spectral background.

The oxidation states of activated V_2O_5 are studied by EELS: the sample milled for 3h contains unreduced (oxidation state 5) and reduced small particles (oxidation state down to 4.3), where vanadium can be in V^{5+} and V^{4+} state. Milling up to 5 h induces further reduction of vanadium (down to 4.1), but unreduced particles can still be found. In the sample milled up to 10 h, however, all the studied particles are reduced; the highest oxidation state is 4.6 and the lowest oxidation state is 3.7, i.e., particles appear which contain vanadium only in the V^{4+} and V^{3+} states. Prolonging the milling up to 20 h gives rise to the total reduction of V^{5+} state and only V^{4+} and V^{3+} states can be detected.

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References

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