Mechanochemical Synthesis of Nanosized Fe_2O_3-TiO_2 Mixed Oxides

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Fe_2O_3-TiO_2 nanosized mixed oxides with Fe_2O_3/TiO_2 ratio of 1:1 and 3:1 were synthesized using mechanical activation of hematite and rutile in high-energy ball mill in argon atmosphere and varying the milling procedure. Similarly, mechanochemically activated pure hematite and rutile materials were also prepared as references. The obtained materials were characterized by low temperature nitrogen physisorption, powder X-ray diffraction, infrared and diffuse-reflectance spectroscopies and TPR with hydrogen. Methanol decomposition was used as a test reaction for the catalytic activity.

The reflection lines in the XRD patterns of all activated samples were broad and of low intensity indicating the presence of nanocrystalline particles with average size of 30-50 nm. The prolonging of the milling time at low milling intensity facilitates the formation of materials with fine particles and high specific surface area. The infrared spectra of the obtained bi-component materials represented the typical absorption bands of pure α-Fe_2O_3 only in the case of the sample with Fe_2O_3/TiO_2 ratio=3:1. The decrease in intensity of these bands for the other samples is an evidence for increased interaction between TiO_2 and hematite nanoparticles. The UV-vis spectra of all bi-component samples were superposition of characteristic bands of rutile and hematite spectra. The presence of isopropyl alcohol during the mechanochemical activation strongly affects the position of the absorption bands near 350 nm, which is assigned to $^6A_1 \rightarrow {}^4E(^4D)$ ligand field transitions of Fe^{3+} ions. The shift of the band to higher wavelength is an indication for the changes in their environment probably due to the formation of a new bi-component oxide phase. This suggestion is also confirmed by the TPR results.

Methanol decomposition is observed above 525-550 K, CO, methane and dimethyl ether being the main registered carbon-containing products. The highest conversion combined with high selectivity to CO (above 85 %) was registered for the bi-component sample, prepared by mechanochemical activation in isopropanol.

The physicochemical and catalytic results reveal a facilitated effect of the wet milling with isopropanol for mechanochemical preparation of Fe_2O_3-TiO_2 mixed oxides.

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