

Structural Investigation of Combustion Synthesized Cu/CeO₂ Catalysts by EXAFS and Other Physical Techniques

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The structure and chemical environment of Cu in Cu/CeO₂ catalysts synthesized by the solution combustion method have been investigated by X-ray diffraction (XRD), transmission electron microscopy (TEM), electron paramagnetic resonance (EPR) spectroscopy, X-ray photoelectron spectroscopy (XPS), cyclic voltammetry (CV), and extended X-ray fine structure (EXAFS) spectroscopy. High-resolution XRD studies of 3 and 5 atom % Cu/CeO₂ do not show CuO lines in their respective patterns. The structure could be refined for the composition Ce_{1-x}Cu_xO_{2-δ} (x = 0.03 and 0.05; δ = 0.13 and 0.16) in the fluorite structure with 5–8% oxide ion vacancy. High-resolution TEM does not show CuO particles in 5 atom % Cu/CeO₂. EPR as well as XPS studies confirm the presence of Cu²⁺ species in the CeO₂ matrix. Redox potentials of Cu species in the CeO₂ matrix are lower than those in CuO. EXAFS investigations of these catalysts show an average coordination number of 3 around the Cu²⁺ ion in the first shell at a distance of 1.96 Å, indicating the O²⁻ ion vacancy around the Cu²⁺ ion. The Cu–O bond length also decreases compared to that in CuO. The second and third shell around the Cu²⁺ ion in the catalysts are attributed to – Cu²⁺ – O²⁻ – Cu²⁺ at 2.92 Å and – Cu²⁺ – O²⁻ – Ce⁴⁺ – at the distance of 3.15 Å, respectively. The present results provide direct evidence for the formation of a Ce_{1-x}Cu_xO_{2-δ} type of solid solution phase having –□– Cu²⁺–O– Ce⁴⁺ – kind of linkages.

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