

Synthesis of Single-phase Cobalt Oxide and Mixed-metal Cobalt Oxide Nanoparticles via a Surfactant-free Approach

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Introduction

Transition metal crystallites and their metal oxides in the nanometer range are the matter of a broad range of interdisciplinary research in fields like recording media, biomedicine, and catalysis. The controlled synthesis of nanoparticles with defined shape, size and a narrow size distribution without a size selection process is challenging and only small amounts of well-defined nanocrystals are achieved in conventional chemical synthesis routes [1]. Widely applied colloidal synthesis routes via the thermal decomposition of organometallic compounds [2] usually require surfactants aiding and stabilising the uniform growth of the crystallites. The development of surfactant-free synthesis routes for controlled nanoparticle preparation is highly desirable, especially for their application in the field of nano-catalysis. Benzyl alcohol is the most applicable solvent for surfactant-free nonaqueous routes. It reacts with a broad band of precursors [3] and has been applied in the synthesis of highly crystalline Co_3O_4 crystallites with narrow size distributions with mean sizes in the range of 3-11 nm [4,5]. Furthermore, the preparation route has also been applied for mixed-metal cobalt oxide nanoparticles [6,7].

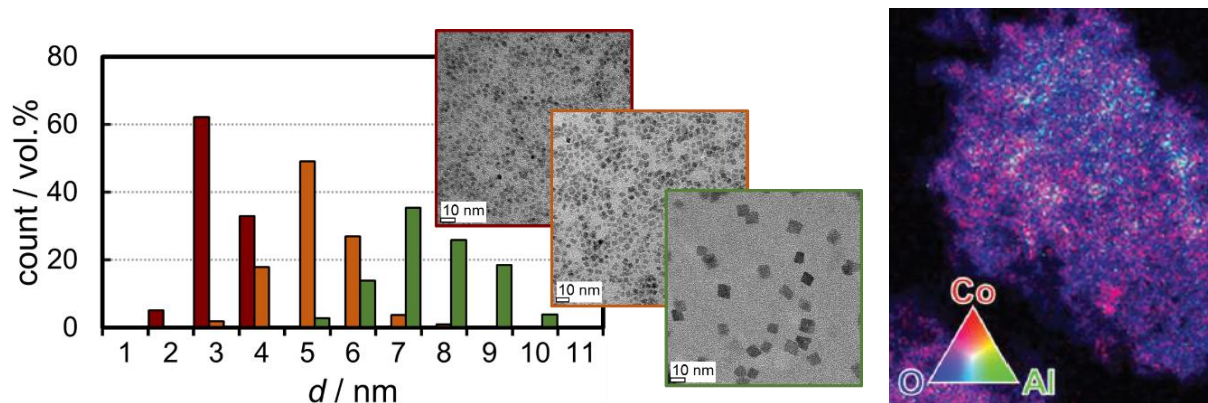


Fig. 1: (left) Size distributions and TEM micrographs of Co_3O_4 nanoparticles [5] and (right) HRSTEM-EELS micrograph of a cobalt aluminate particle [6].

Experimental

Cobalt oxide, other metal oxides (e.g. Cu, Fe, In), and mixed-metal cobalt oxide nanoparticles were prepared via heat treatment of dissolved metal acetates or alkoxides and ammonium hydroxide in benzyl alcohol [4-7]. The as prepared nanoparticles were characterised via transmission electron microscopy (TEM), X-ray diffraction (XRD) and employed in the synthesis of supported catalysts by mixing nanoparticles and common support materials during ultrasonication.

Results

At first, previously reported parameters for controlling the size of Co_3O_4 nanoparticles were confirmed, namely the amount of precursor, the synthesis temperature, and the amount of ammonia solution. Cobalt nanoparticles were successfully prepared in the range of 2.5-9.0 nm. Further, a series of other metal oxide nanoparticles has been synthesized by replacing the cobalt acetate precursor with other metal acetates. Subsequently, mixed-metal cobalt oxides were prepared via a simultaneous addition of cobalt and other metal acetates in a one-pot synthesis. TEM and XRD suggest a high crystallinity and narrow size distributions, while XRD confirmed the formation of mixed-metal oxides for some instances.

References

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