Synthesis of nanocrystalline Co_3O_4 nanopowders via self-propagation combustion reaction

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Over the past two decades, a considerable effort has been devoted to research of the p-type semiconducting materials, which found diverse applications in industry. As the p-type material with a spinel AB_2O_4 structure and direct optical band gap of 2.19 eV, cobalt oxide, Co_3O_4 has drawn special interests in gas-sensing and solar energy absorption research community. At the same time, it has found the usage as an electrode material in lithium ion batteries, catalyst, ceramic pigment, field-emission and magnetic material. A powerful strategy to improve the Co_3O_4 performances in some of the mentioned applications is the utilization of a nanocrystalline powder with a high surface to volume ratio, which can be successfully produced by self-propagation combustion reaction.

In this work, cobalt nitrate (oxidizer) and glycine (fuel) of required amounts were dissolved in the minimum amount of deionized water to obtain a clear solution. According to the rules of propellant chemistry, the glycine/metal ion ratio was adjusted to provide stoichiometric or fuel-lean conditions of the redox reaction. The sols were dried at 80 ^{o}C to obtain the gels, and then subjected to the rapid heating, where the gels auto-ignited at approximately 250 ^{o}C (depending on the amount of the fuel) and spontaneously underwent a smouldering combustion with evolution of large amounts of gases, subsequently forming a voluminous nanosized Co-oxide powder.

It is worth to mention that the dual role of the glycine (chelating agent and fuel) in this synthesis was of great importance in preventing the precipitation of the solution during rapid heating, and in liberation of the required energy for the synthesis of the Co_3O_4 powder with desired Co^{+2}/Co^{+3} oxidation state, necessary for its further use in production of gas sensors and catalysts. The processes involved in formation of the pure Co_3O_4 powder and especially the exothermicity of the reaction, were followed by simultaneous thermal analysis (thermogravimetric and differential thermal analysis coupled with evolved gas analysis). According to the X-ray diffraction analysis the phase-pure Co_3O_4 was obtained only when the precursor powder was prepared from the fuel-lean redox reaction. The field emission scanning electron micrographs revealed the spongy aspect of the calcined powder, where small primary particles formed the agglomerates. The transmission electron microscopy was used to get deeper insight in the particle size and purity of the as prepared nanosized powders. The formation of the single Co_3O_4 powder from the solution to the combusted powder was also analysed by Fourier transformed infra-red spectroscopy.

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