

# **Synthesis, Characterization and Properties of Transition Metal Oxide and Hydroxide Nano materials**

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## **Abstract**

Material synthesis is a constantly evolving area of research and thus recognizing the limitations of current routes for the synthesis of a range of transition metal oxides is important in devising newer strategies for their growth. Transition metal oxides, as a group of advanced functional materials, exhibit several unique and interesting properties as a result of their small dimensions. Morphology of the products, their surface area, lattice defect structure and nature of the surface directly affect the practical capacity of oxide nanomaterials in different applications. The present work intends to develop common strategies, applicable to the synthesis of more than one binary transition metal oxide or its hydroxide, in solutions using hydrothermal and low temperature aqueous chemical growth routes. This includes surface modification of nanoparticles with a common modifier to tailor the surface properties, achieve control over the phase and morphology of nanostructures by varying a single experimental parameter, or nucleating them on substrates from a common solution under similar conditions. Five different oxide systems that include ZnO, MnO<sub>2</sub>, NiO and Ni(OH)<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and CuO were at the focus of research.

Two novel approaches were used to produce different ZnO nanostructures. First, surface modified, fluorescent ZnO nanoparticles with sizes in the range of 11-15 nm were prepared at 80°C in 2 hours time. The modified nanoparticles emit strongly in the green region of the

spectrum; a property that proved useful in imaging biological tissues. Second, a seedless aqueous chemical growth route was used to grow first, dense single crystalline ZnO nanorod arrays on 3D nickel foam substrates. Growth conditions, both for nanoparticles and nanorod arrays, were optimized by varying the concentration of starting materials, growth time, and synthesis temperature.

Next, crystalline  $\alpha/\beta$ -Ni(OH)<sub>2</sub> nanosheets, nanobelts and nanorods were prepared by hydrothermal treatment of amorphous precipitates obtained at different pH values. The morphology and crystal structure of the products was controlled simply by adjusting the pH value at precipitation. Thermal annealing of the hydroxides yielded cubic phase NiO with morphologies similar to their hydroxide precursors. Also, a simple method is presented to grow honeycomb,  $\beta$ -Ni(OH)<sub>2</sub> thick films on 3D nickel foam substrates at 80°C. The porous honeycomb network with regular pores is built from seamlessly connected nanowalls, approximately 10-20 nm thick. Again, air annealing of the as-prepared films resulted in honeycomb NiO films.

The procedure used to grow ZnO and Ni(OH)<sub>2</sub> films on the 3D nickel foam substrate was successfully employed to grow thick CuO films. A thick coating of 3-4  $\mu$ m, consisting of interlaced CuO nanosheets, covered well the entire foam substrate. The sheets were rough at the edges with a mean thickness in the range of 20-30 nm. Due to the faster transformation kinetics of [Cu(OH)<sub>4</sub>]<sup>2-</sup> to CuO in aqueous solutions, all the precursor material tend to nucleate heterogeneously over a short period, extending the growth time had therefore little effect on the thickness of the films formed.

For the synthesis of  $\text{MnO}_2$ , a simple hydrothermal route was used to grow urchin-like  $\gamma$ - $\text{MnO}_2$  nanostructures by a direct reaction between  $\text{MnSO}_4$  and  $\text{KClO}_3$  under mild conditions. The urchin-like spheres were composed of nanowires with diameters in the range 40-70 nm. The urchins were subsequently transformed to  $\alpha$ - $\text{MnO}_2$  nanowires on prolonging the hydrothermal dwell time. A possible rolling-broken-growth mechanism was suggested for the self-assembly of  $\text{MnO}_2$  nanowires into urchins and, Ostwald ripening was responsible for their subsequent phase transformation. The urchins when tested by cyclic voltammetry for their charge storage ability exhibit excellent rate capability with a specific capacitance of  $225 \text{ F g}^{-1}$  at a scan rate of  $5 \text{ mVs}^{-1}$ .

Surface modified  $\alpha$ - $\text{Fe}_2\text{O}_3$  nanoparticles capped by Tween-80 were prepared by the hydrothermal treatment of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  at  $200^\circ\text{C}$ . The spherical nanoparticles possessed good crystallinity with an average crystallite size of 21 nm. The presence of Tween-80 on the surface of  $\alpha$ - $\text{Fe}_2\text{O}_3$  was confirmed by FTIR and Mossbauer analysis. The surfactant was effective in controlling the particle shape and restricted the particle growth to a narrow range around 40-60 nm as observed by scanning electron microscopy. The  $\alpha$ - $\text{Fe}_2\text{O}_3$  nanoparticles obtained without Tween-80 were irregular in shape with a wide size distribution in the range of 150-300 nm.