Nanocrystalline CuAlO₂ powder by the nitrate-citrate combustion route

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The copper aluminate (CuAlO₂) with the delafossite structure has recently drawn attention as a promising p-type semiconducting material. It can be used as a transparent conducting oxide in the field of invisible electronics for production of different optoelectronic devices, but also as a catalyst for conversion of the solar power to hydrogen energy, room temperature ozone sensor, or thermoelectric. The performance of CuAlO₂ in some of the mentioned applications could be optimized by increasing the surface area and decreasing its particle size. For that purpose, the nanocrystalline CuAlO₂ powder was synthesised by the self-sustained combustion route.

Citric acid was introduced both as the chelating and reducing agent or fuel. The citric acid/metal ion ratio was adjusted to provide fuel-lean, stoichiometric, and fuel-rich conditions of the redox reaction. Equimolar amounts of copper and aluminium nitrates were dissolved in deionized water. Then, the citric acid was added. The solution was agitated at room temperature for 1 h, and then neutralized to increase the efficiency of the chelating agent. After several hours of drying in the temperature range $60\,^{\circ}\text{C} - 80\,^{\circ}\text{C}$, the sol turned to a homogeneous viscous gel. During the rapid heating, the gel auto-ignited at approximately $250\,^{\circ}\text{C}$ and spontaneously underwent smouldering combustion, forming the grey-black voluminous precursor powder.

According to the X-ray diffraction analysis the phase pure delafossite was obtained only when the precursor powder was prepared from the stoichiometric redox reaction, and after the calcination at 920 °C for 4 h in Ar atmosphere. The field emission scanning electron micrographs revealed the cauliflower aspect of the calcined powder, where small primary particles formed the agglomerates. The formation of the phase pure CuAlO₂ powder was also followed by Fourier transformed infrared spectroscopy and confirmed by selective area diffraction analysis.