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A combined spectroscopic and structural characterisation of crystalline nanostructured manganites prepared by a green and low temperature hydrothermal route

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The low temperature synthesis of nanostructured oxides in an aqueous medium is a topic of great interest from the environmental point of view. Among metal oxides, manganites ($M_xMn_yO_z$; M = transition or alkali metal) are endowed with interesting magnetic, electrical and catalytic properties. In this framework, zinc ($ZnMn_2O_4$ and $ZnMnO_3$) and copper ($CuMnO_2$) manganites were synthesised through an easy, reproducible and low-temperature hydrothermal route starting from an aqueous suspension of coprecipitated metal oxalates. The powders were obtained at the low temperature of 180°C and no further thermal treatment or purification were needed. The cubic spinel $ZnMnO_3$ is of particular interest, as very few works are available where the compound was obtained as a pure crystalline phase. The thorough characterisation of the obtained materials was accomplished by a combined approach of structural and spectroscopic methods, in part based on the use of synchrotron radiation. In particular, the structure of the compounds was investigated at the long- and short-range order by the combined use of XRD and XAS. These analyses yielded evidence of zinc deficiency in cubic spinel $ZnMnO_3$. Furthermore, XPS and XAS revealed the presence of different Mn oxidation states on the surface and in the bulk, respectively, and allowed to determine the local Mn structure. Synchrotron-based time-resolved SAXS experiments were also performed in order to study the crystallization mechanism of the manganites under simulated hydrothermal conditions. To investigate the morphology of the manganites, TEM/SEM observations were carried out on selected samples. As far as functional characterisations are concerned, SQUID was used to investigate the magnetic properties.