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Synthesis and morphological study of chromium substituted Zn–Mn ferrites nanostructures via sol–gel method

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ABSTRACT

Nanocrystalline $ZnMn_{1-x}Cr_xFeO_4$ ($1.0 \geq x \geq 0$) ferrites were prepared by sol–gel route. X-ray diffraction (XRD) method was used to confirm the formation of single phase cubic spinel lattice for all the composition. The lattice parameter (a) shows a decreasing trend with the increase in Cr content. In all the studied compositions, spherical crystalline nanoparticles of about 30 nm size were observed by transmission electron microscopy (TEM) technique. The elemental analysis as obtained from EDAX is in close agreement with the expected composition from the stoichiometry of reactant solutions used. Infrared spectroscopic studies revealed two main absorption bands in the range of 400–800 cm^{-1} arising due to tetrahedral (A) and octahedral (B) stretching vibrations. On substitution of Cr^{3+} in Mn^{3+} site, the stretching frequency of the octahedral site increases smoothly but that of the tetrahedral site is seen to be unaltered. The detailed results of XRD, SAED and infrared spectroscopy have been discussed so as to bring out the role of chromium substitution in determining structural properties of Zn–Mn ferrites.

1. Introduction

Mixed-metal oxide nanoparticles have been intensively studied in the last decade due to their unusual physical and chemical properties owing to their extremely small size, large specific surface area and number of promising applications. Among the various classes of nanomaterials, metal oxides are the common, most diverse and possess richest class in terms of physical, chemical and structural properties. As a result, numerous applications of metal oxides, such as fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, dielectrics, lasers, magnets and catalysts have been discussed [1–13].

Recently, considerable effort has been made on the surface modification of nanoparticles and the preparation of different types of metal oxides. Various methods are available for the synthesis of metal oxides, such as microwave refluxing [14], sol–gel [15,16], hydrothermal [17,18], co-precipitation [19,20], citrate–gel [21], spray pyrolysis [22], etc. The selection of appropriate synthetic procedure often depends on the desired properties and final applications. Among these synthesis techniques, sol–gel autocombustion method has several advantages over others for preparation of nanosized metal oxides as the process begins with a relatively homogeneous mixture and involves low temperature conditions resulting in a uniform ultrafine porous powders [23]. In our previous work [24,25] this method was employed to obtain improved powder characteristics, better homogeneity and narrow particle size distribution, thereby influencing structural, electrical and magnetic properties of spinel ferrites [26]. In this communication, we report preparation of nanosized chromium substituted Zn–Mn ferrites by sol–gel autocombustion method. The structural and morphological properties investigated by X-ray diffraction (XRD), TEM, SAED, energy dispersive X-ray spectroscopy and FTIR spectroscopy have also been discussed in details.

2. Experimental technique

Analytical grade chromium nitrate [$Cr(NO_3)_3{\cdot}9H_2O$], iron nitrate [$Fe(NO_3)_3{\cdot}9H_2O$], zinc nitrate [$Zn(NO_3)_2{\cdot}6H_2O$], manganese nitrate [$Mn(NO_3)_2{\cdot}4H_2O$] and citric acid [$C_6H_8O_7{\cdot}H_2O$] were used to prepare $ZnMn_{1-x}Cr_xFeO_4$ (where $x = 0.0$, 0.25, 0.50, 0.75 and 1.0) by sol–gel method. Metal nitrates and citric acid were dissolved in minimum quantity of deionized water with 1:1 molar ratio. The pH of the solution was adjusted to about 9.0–9.5 using ammonia solution. The solution was transformed to dry gel on heating to 353 K. On further heating the dried gel burnt in a self propagating combustion manner until all the gel was completely converted to a floppy loose powder. The as burnt precursor powder was then sintered at 973 K for 8 h. The sintered powders were granulated using 2% polyvinyl alcohol as a binder and were uniaxially pressed at a pressure of 8 ton/cm^2 to form pellets. These pellets were gradually heated to about 773 K to remove out the binder material.

The phase formation of the sintered samples were confirmed by X-ray diffraction studies using a Philips PW-1710 X-ray diffractometer using CrKα radiation ($\lambda = 2.2897$ Å) in a θ–2θ geometry at standard atmospheric conditions. FTIR study was used to indicate the vibrational modes in the samples. The morphology and particle size analysis was carried out on a transmission electron microscope

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