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ORIGINAL ARTICLE

A first report on the crystal structure of hydrazine based solid solution precursor, $Mn_{0.61}Zn_{0.39}(OOCCH{=}NNH_2)_2(H_2O)_2$

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KEYWORDS

Hydrazoneglyoxylate;
Aqueous condensation;
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Mixed metal oxide;
Crystal structure

Abstract A novel mixed metal complex, $[Mn_{0.61}Zn_{0.39}(OOCCH{=}NNH_2)_2(H_2O)_2]$ has been synthesized and characterized by hydrazine and metal analyses, CHN analyses, infrared spectra, simultaneous TG-DTA and single crystal X-ray diffraction studies. The structure of the complex has been determined by X-ray single crystal study which clearly reveals that the octahedral site is occupied by two metal ions (viz., Mn and Zn) around which two hydrazoneglyoxylate ligands ($OOCCH{=}NNH_2$) coordinate in a bidentate chelating fashion. Two water molecules occupy the remaining two other sites of a slightly distorted octahedron with C_2 axis of symmetry. The infrared spectrum reveals the monodentate coordination behavior of carboxylate ions and coordination of water molecules. The simultaneous TG-DTA traces are in accordance with the formation of mixed metal oxide (spinel oxide) as an end product. The high resolution scanning electron microscopy (HRSEM) images show the presence of irregularly shaped agglomerated particles of sub-micron size. The zeta potential value for mixed metal oxide is −33.1 mV, which indicates the stability of the oxide.

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1. Introduction

Solid solutions are of great importance in the field of coordination chemistry and material science. Several hydrazine based solid solutions have been reported in the literature which were exploited as precursors for mixed metal oxides such as cobaltites, ferrites and manganites [1–6]. Though the formation and structure of such solid solutions have been assigned only on the basis of spectral, thermal and analytical studies, the crystal structure of any such mixed metal complexes has not

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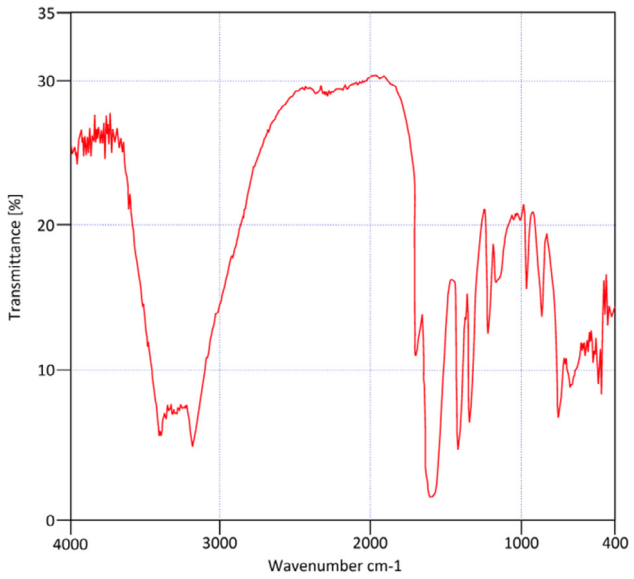

Fig. 1 Infrared spectra of $Mn_{0.61}Zn_{0.39}(OOCCH{=}NNH_2)_2(H_2O)_2$.

been reported so far. This is due to the difficulty in obtaining and isolating the single crystals of mixed metal complex suitable for X-ray single crystal studies. Hence, the compositions assigned were on the basis of analytical, spectral and thermal studies and also on the basis of the spinels formed during their pyrolysis. The X-ray crystal structure determination is the only way of predicting the exact composition and geometry of such complexes which has not been successfully carried out for such solid solutions. The purity and compositions of the spinels obtained depend only on the composition of the solid solutions. The particle size and potential stability of the mixed metal oxides were widely used in biological and surface chemistry [7,8].

Recently simple metal hydrazoneglyoxylate hydrates have been prepared by the aqueous condensation of glyoxylic and hydrazine hydrate in the presence of metal ions [9]. These complexes were found to be isomorphic and hence the investigations on the synthesis and structural characterization of mixed metal complexes (solid solutions) have been initiated. For the first time in hydrazine chemistry a single crystal of hydrazoneglyoxylatodiaquo mixed metal complex containing Mn^{2+} and Zn^{2+} in 0.61:0.39 ratio has been isolated and characterized. In this paper, we report the preparation and structure of $Mn_{0.61}Zn_{0.39}(OOCCH{=}NNH_2)_2(H_2O)_2$ and also its spectral, fluorescence emission and thermal properties. We also report HR-SEM, particle size and potential stability of the mixed metal oxide prepared by the combustion of the solid solution precursor.

2. Experimental section

2.1. Materials and methods

All the chemicals and solvents were purchased from S.D. Fine Chemicals, Mumbai, India. The solvents were distilled before use and double distilled water was used for the preparation and analyses of the complex.

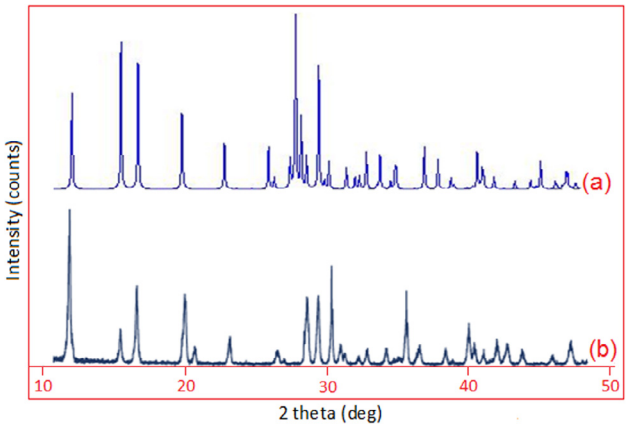

Fig. 2 Powder X-ray diffraction patterns of $Mn_{0.61}Zn_{0.39}(OOCCH{=}NNH_2)_2(H_2O)_2$ (a) stimulated (b) experimental.

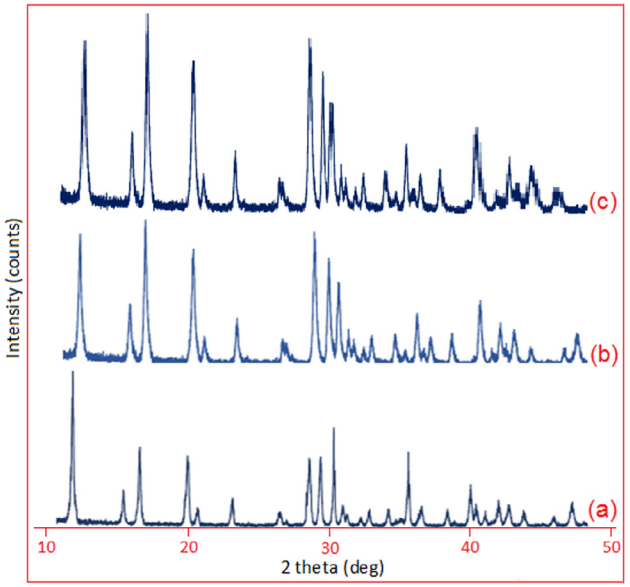

Fig. 3 Powder X-ray diffraction patterns of (a) $Mn_{0.61}Zn_{0.39}(OOCCH{=}NNH_2)_2(H_2O)_2$ (b) $Zn(OOCCH{=}NNH_2)_2(H_2O)_2$ (c) $Mn(OOCCH{=}NNH_2)_2(H_2O)_2$.

The hydrazine content was determined by volumetric analysis using a 0.025 M KIO_3 solution under Andrew's condition. When concentrated HCl is added to the aqueous suspension of complex, the complex decomposes to give hydrazine hydrochloride from which the quantity of hydrazine was determined volumetrically [10–12]. The C, H and N analyses for the complex was performed using Perkin-Elmer-1240 CHN analyzer. The metal contents were determined by Inductively Coupled Plasma-Optical Emission spectroscopy (ICP-OES) using Perkin Elmer Optima 5300-DV ICP-OES. Infrared spectrum of the complex was recorded (KBr disc, 400–4000 cm^{-1}) a Bruker Alpha spectrometer. The simultaneous TG-DTA of the sample in air was recorded using a SWI TG/DTA 6200 thermal analyzer using about 5 mg of the sample with the heating

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