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Heat treatment effects on structural and dielectric properties of Mn substituted $CuFe_2O_4$ and $ZnFe_2O_4$ nanoparticles

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ABSTRACT

Manganese substituted copper and zinc ferrite nanoparticles were synthesized by an auto-combustion technique using metal nitrates and urea. The nanoparticles were characterized by XRD, SEM, EDX, and TEM techniques. The effect of annealing temperature on structural and dielectric properties of Mn substituted spinel ferrite nanoparticles was analyzed. The presenting elements in the prepared samples are recorded by EDX. TEM analysis clearly showed the particles are in the nanometer range. The dielectric loss and dielectric constant have been measured in the frequency range of 100 kHz–5 MHz. The variation in structural and dielectric properties of the prepared and annealed samples are discussed.

1. Introduction

Development of magnetic nanoparticles has been intensively pursued because of their technological and fundamental scientific importance [1]. Several interesting phenomena have been discovered in recent years based on the studies of nanoparticles of magnetic and non-magnetic materials [2]. Various physical properties of the magnetic nanoparticles are greatly influenced by the distribution of cations among the sublattices, nature of grain, grain boundaries, voids, inhomogeneities, surface layers, chemical defects, oxygen deficiency and contacts, etc. These magnetic nanoparticles have received great attention as a result of their magnetic and electronic properties. Such magnetic nanoparticles are currently used in ferrofluids, microwave devices, bioprocessing [3–8], etc. The magnetic properties of the nanoparticles can be varied systematically by changing the identity of the divalent cations. There are several methods for synthesizing nanosized magnetic spinel ferrite particles, such as co-precipitation [9–12], sol–gel [13], standard double-sintering method [14], microwave-induced combustion [15], microwave sintering method [16], sol–gel auto-combustion method [17], reverse micelle reaction process [18], and evaporation method [19]. The present attempt is made on manganese substituted copper and zinc ferrite nanoparticles by an-auto combustion method using urea as a fuel. The study mainly focuses on the effect of heat treatment on the structural and dielectric properties of $CuFe_2O_4$ and $ZnFe_2O_4$ nanoparticles. The results indicate that the process is convenient, inexpensive, environment friendly and efficient for preparation of Mn substituted metal nanoparticles with the size of about ~9–45 nm.

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2. Experimental technique

2.1. Synthesis process

Nanocrystalline manganese doped cupper ferrite has been prepared with the chemical formula $Mn_{0.4}M_{0.6}Fe_2O_4$ (M = Cu and Zn) by an auto-combustion technique. The analytical grade manganese nitrate [Mn $(NO_3)_2{\cdot}6H_2O$], copper nitrate [Cu $(NO_3)_2{\cdot}6H_2O$], ferric nitrate [Fe $(NO_3)_3{\cdot}9H_2O$], zinc nitrate and urea [CO $(NH_2)_2$] were used as raw materials. Subsequently, 0.4 M of manganese nitrate, 0.6 M of metal nitrate, 2 M of ferric nitrate and urea were dissolved in deionized water to form mixed solution, using a magnetic stirrer. Then the mixture was heated at 160 °C to dehydrate until self-ignition takes place. Being ignited in air at room temperature, the dried gel burnt in a self propagating combustion, giving rise to the evolution of large amount of gases and producing a dry and loose ferrite powder. Throughout the process no pH adjustment was made. The influence of heat treatment on particle size and dielectric properties were studied, a portion of the as-burnt ferrite powders was sintered at 600 °C and 900 °C for 5 h.

2.2. Equipment used for characterization

The ferrite powders were subjected to XRD analysis with Rigaku X-ray diffraction unit (Model ULTIMA III) to explore the structural properties. Scanning electron microscope (SEM with EDX) was used to examine the particle morphology, using a JEOL 5600LV microscope at an accelerating voltage of 10 kV. High resolution transmission electron microscopy (HRTEM) and selected-area electron diffraction (SAED) was recorded on a Technai G20-stwin using an accelerating voltage of 200 kV. The dielectric properties of as-burnt and annealed samples were measured by Digital LCR meter (Model TH2816A) in the frequency range from 100 kHz to 5 MHz.

3. Results and discussion

3.1. Structural analysis

The XRD pattern to determine the phase compositions of the as-burnt and annealed samples of $MnCuFe_2O_4$ and $MnZnFe_2O_4$ nanoparticles are shown in Fig. 1. The reflection peaks correspond to the characteristic interplanar spacing between (220), (311), (222), (400), (422), (511) and (440) planes of the spinel ferrite with a cubic symmetry, demonstrating the formation of Mn substituted MFe_2O_4 (M = Cu and Zn) ferrites as per JCPDS data. XRD pattern of the as-burnt powder contains no secondary peaks, which suggests that the sample has pure spinel structure. From the results, the Mn–Cu and Mn–Zn ferrite phase contained some impurity peaks, which are due to decomposition of the ferrites to α-Fe_2O_3 phase, above the annealing temperature of 500 °C [20–22]. The diffraction peaks become narrower and sharper suggesting that there is an increase in particle size upon annealing. The average particle size of prepared powder has been calculated using Debye–Scherrer formula [23].

$$t = \frac{0.9\lambda}{\beta \cos\theta} \tag{1}$$

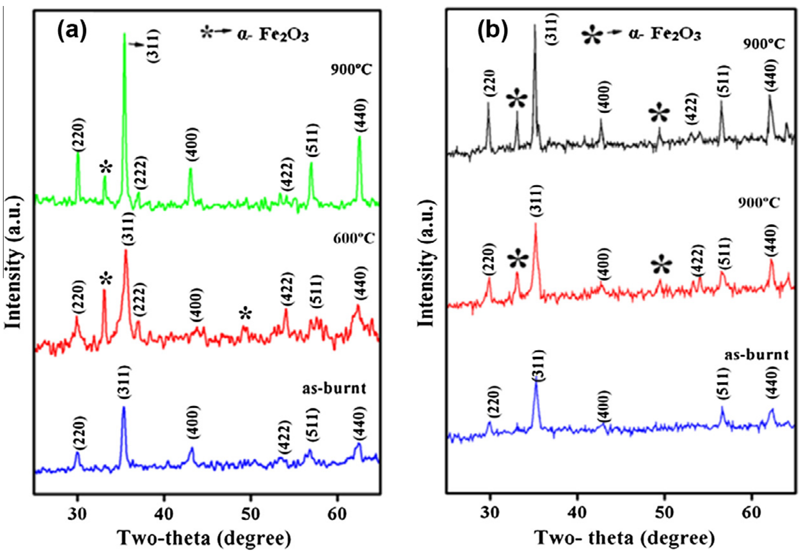

Fig. 1. XRD pattern of Mn substituted copper and zinc ferrite nanoparticles.