Original Article

Study on Synthesis Characterization and Hydrophilic properties of CdFe$_2$O$_4$ Nanoparticles

Sachin V. Bangale

Department of Chemistry, Gopinath Mahadev Vedak College of Science Tala-Raigad 402111(M.S.) India

E-Mail: bangale_sv@rediffmail.com
Phone No.02140-269008, Fax No. 02140-269008

Received 02 August 2013; accepted 21 August 2013

Abstract

Nano structured CdFe$_2$O$_4$ is synthesized by combustion method using glycine as a fuel. The synthesized material was characterized by TG-DTA, XRD, SEM and TEM. The average particle size of the nanomaterial CdFe$_2$O$_4$ calculated from XRD was found in the range 64 nm. The superhydrophilicity of the sintered oxides was investigated by wetting experiments, by the sessile drop technique, were carried out at room temperature in air to determine the surface and interfacial interactions.

Keywords: TG/DTA, XRD, CdFe$_2$O$_4$, combustion method.

1. Introduction

The spinel compounds AB$_2$O$_4$, where A and B are metal cations and X is an anion have been extensively studied for their magnetic properties [1-3]. Due to the unique physical and chemical properties, synthesis and application of nanoparticles is the focus of intense research. These materials are very attractive in view of both, their scientific and technological importance. Nanocrystalline spinel ferrites with the common formula MFe$_2$O$_4$ (M = Ni, Zn, Mn, Co, Mg etc.) are the most significant magnetic materials [4]. The spinel structure belong to space group Fd3m. The cubic unit cell is formed by 56 atoms, 32 oxygen anions dispersed in a cubic close packed structure, and 24 cations occupying 8 of the 64 tetrahedral site (A site) and 16 of the 32 octahedral site (B site) [5]. Nickel ferrite powder, one of the very important ferrite materials has been considered for many application such as high density magnetic storage media, MRI contrast agent, colour imaging, ferro-fluids, high frequency devices, magnetic refrigerators, gas sensor, magnetic fluids, photomagnetic materials, site-specific drug delivery and microwave devices [6-8]. The properties of the synthesized materials are influenced by the composition and microstructure, which are sensitive to the preparation methodology used in the synthesis. Various methods such as citric acid combustion methods [9], sol-gel autocombustion method[10], organic gel-thermal decomposition method [11], hydrothermal method [12], co-precipitation method [13], gel-assistant hydrothermal route [14], thermolysis [15], wet chemical co precipitation technique [16], self-propagating [17], microemulsion [18] and microwave synthesis [19] have been developed to prepare nanocrystallite nickel ferrites.

We report here the synthesis of nickel ferrites nanoparticles through combustion method which is a unique combination of the ignition and the chemical gelatine processes. This method has the advantages of simple preparation, cost effective and gentle chemistry route resulting in ultra fine and homogeneous powder. The ability to obtain sonephage nickel ferrites magnetic nanoparticle with controllable particle size and size distribution improves its adequacy in a wide range of technological application. CdFe$_2$O$_4$ nanoparticles are prepared by combustion method, the structural, thermal, morphological and hydrophilic properties are investigated.

2. Method and Material

2.1. Preparation of materials

For the present study, polycrystalline CdFe$_2$O$_4$ powder was prepared by combustion route [20-22] using glycine as fuel. The materials used as precursors were Cadmium nitrate hexahydrate Cd (NO$_3$)$_2$.6H$_2$O, Fe (NO$_3$)$_3$6H$_2$O Iron nitrate hexahydrate (all these were procured from A.R. Grade of Qualigen) and Citric acid (Nuclear band). glycine possesses a high heat of combustion. It is an organic fuel and provides a platform for redox reactions during the course of combustion. Initially the Cadmium nitrates, Iron nitrates and glycine are taken in the 1:1:4 stoichiometric amounts and dissolved in 250 ml beaker slowly string with glass rod clear solution was obtained. Solution formed was evaporated on hot plate in temperature range 70°C to 80°C gives thick gel. The gel was kept on a hot plate for auto combustion and heated in the temperature range 170°C to
The nanocrystalline CdFe₂O₄ powder was formed within few minutes and sintered at about 600°C for about 4 hours got shining powder of nanocrystalline CdFe₂O₄ as shown in following sheet [20-23].

2.2 Characterization technique

The prepared CdFe₂O₄ samples were characterized using TG/DTA thermal analyzer (PERKIN ELMER, USA), X-ray diffract meter (RIGAKU MINIFLEX-II) using Cu–Ka radiation, Scanning Electron Microscope (FEI QUANTA 200). Transmission electron microscope operating at 200 kV.

3. Results and Discussion

3.1 TGA Analysis

Figure 1 the first three intervals are entwined from 40°C to 360°C with broad endothermic peaks and a weight loss of 20%. These are attributed to the evaporation of residual water and burning of residual organic materials. The second from 400°C to 600°C with a rapid weight loss of 50% and a broad exothermic peak around 600°C, this is attributed to decomposition of the organic compounds. The synthesized powder was almost stable from the 600°C.

3.2 X-Ray Analysis

The XRD pattern of the mixed precursor calcined in air at 600°C for 4 h is shown in Figure 3. It exhibits the diffraction peaks at 20 values of, 35.57°, 37.16°, 43.19°, 57.11°, 62.82° and 75.45° at 600°C which were attributed to the formation of CdFe₂O₄ spinel structure in the calcined material. The calculated lattice parameter a = 8.3275 Å was in good agreement with the reported value for CdFe₂O₄ spinel (a = 8.3275, JCPDS # 74-2081). The crystallite size was calculated by using the Scherrer equation t = Kλ/βcosθ, where t is the average size of the crystallite, assuming that the grain are spherical, K is 0.9, k is the wavelength of X-ray radiation, B is the peak full width at half maximum (FWHM) and θ is the angle of diffraction. The crystalline size of the calcined mixed precursor is found to be 600°C 64 nm.

3.3 EDAX result

TG–DTA curve indicates the phase formation of CdFe₂O₄ is just nearly 600°C therefore EDX carried out only at 600°C represented. Shows the energy dispersive X-ray spectrum of CdFe₂O₄. This was carried out to understand the composition of Cadmium, iron and oxygen in the material. There was no unidentified peak observed in EDX. This confirms the purity and the composition of the CdFe₂O₄ nanomaterial.

3.4 Scanning electron micrograph analysis:

The microstructure of the sintered samples can be visualized from scanning electron microscope (SEM) tool. Figure 3 shown the particle morphology of high resolution, the particle are most irregular in shape with a Nanosize range. Some particles are found as agglomerations containing very fine particles the particles shapes are not defined porous nature and small and large core, spongy pores are seen in the micrograph.

(Fig.1) Thermo gravimetric differential analysis curve of Cadmium ferrite sample.

(Fig.2) Powder XRD pattern of the Cadmium ferrite sample

(Fig.3)SEM images of the self combustion product the powder annealed at 600°C at (a) and (b) high resolution.
3.5 Transmission electron microscopy analysis

The TEM specimens were prepared by placing microdrops of colloid solutions on a carbon film supported by a copper grid. The TEM images of the nanocrystalline CdFe₂O₄ region. The pores are well developed spherical shape with diameter from 70 nm size. The results are correlated with the XRD.

4. Superhydrophilic test

**In to characterization:**

Wetting experiment of synthesized pure cadmium iron oxide evaluated by contact angle measurement were performed by the sessile drop method using an Advanced goniometer (Model110, Ram hart Instrument Co., USA) apparatus and distilled water droplets (0.01ml) were delivered to surface of cadmium iron oxide material at different points.

The wettability nature of our synthesized material is super hydrophilic in the Wenzel because of highly rough surface nature clearly seen from SEM images with consideration given to the surface roughness. Figure 5 (a-b) shows the image of contact angle on rough surface of Cadmium iron oxide material. It seen that contact angle of material is θ =0, hence material in superhydrophilic (θ ≤ 5) may be due to high energy surface and porous nature.

5. Conclusions

Nanocrystalline CdFe₂O₄ has been synthesized by self combustion route. This synthesis route may be used for the synthesis of other metal oxide. The phase formation of the CdFe₂O₄ is investigated by TG-DTA and XRD techniques. The synthesized product shows single phase of inverse spinel structure with an average diameter 64 nm. Elemental calcinated at 600°C in air for 4 h are shown in Figure 5. It indicates the presence of CdFe₂O₄ nanoparticles with 70 nm size which form spherical type of oriental aggregation, agglomeration and polymeric linkage throughout the analysis confirmed by using EDX. Wettability of this material obtained from contact angle goniometer. The contact angle (θ) is zero, which indicates that oxide material was superhydrophilic.

Acknowledgement

The author S. V. Bangale is thankful to Shivaji University Kolhapur for providing the SEM facility. I take this opportunity to thank N.G.Vedak and N.S.Yadav for providing necessary facilities and encouragement during research work.

References

Source of support: Nil; Conflict of interest: None declared