

Study on physico-chemical properties of Cd ferrites synthesized by sol-gel method using different organic fuels

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Abstract

An attempt has been made to study the effect of various reaction conditions on physico-chemical properties of CdFe_2O_4 . Citric acid, urea and polyethylene glycol (PEG) were used as fuels for the combustion. Precursors under consideration along with different organic fuel lead to formation of single phasic, cubic structured cadmium ferrites at different temperatures as depicted from FTIR spectroscopy and XRD patterns with particle size in the range of 18–37 nm revealed from TEM micrographs. The analysis of various parameters of reaction may be used for better understanding of synthesis process and control of microstructure and property of spinel ferrite.

1. INTRODUCTION

Spinel structure constitutes a cubic close packed arrangement with 32 oxygen ions forming a unit cell with 64 tetrahedral (A) sites and 32 octahedral (B) sites. For electrical neutrality of the lattice, the 8 tetrahedral and 16 octahedral sites are occupied by either divalent or trivalent ions with general formula AB_2O_4 units. Distribution of divalent and trivalent cations between the tetrahedral and octahedral sites may be written as the formula $(\text{M}_\delta\text{Fe}_{1-\delta})[\text{M}_{1-\delta}\text{Fe}_{1+\delta}]\text{O}_4$, where 'M' represents a divalent cation and 'δ' is the degree of inversion. The normal spinel structure with $\delta = 1$ has A-sites occupied by divalent cations, while B-sites by trivalent cations. In the inverse spinel structure with $\delta = 0$, divalent cations occupy B-sites, while the trivalent cations are distributed among 'A' and 'B' sites. In a mixed spinel structure, δ ranges between 1 and 0 i.e. both the tetrahedral and octahedral sublattice sites are occupied by divalent and trivalent ions [1].

Bulk cadmium ferrite shows normal spinel structure with negligible magnetic character. Whereas, in the nano range, inverted spinel structure with enhanced magnetic parameters have been reported and therefore, Cd ferrite nanoparticles find applications in the fields of sensors, catalysis and magnetic technologies [2-3].

Various methods viz. ceramic, co-precipitation, sol-gel etc. have been used by the researchers to synthesize ferrite nanoparticles at different temperatures. Ceramic method is economical for the bulk synthesis of ferrites but large sized particles are formed due to aggregation of nanoparticles with low phase purity as high energy milling requires greater energy consumption and also induces structural disorder in the crystallites. In the recent past sol-gel auto combustion method have been preferred over other conventional methods due to synthesis of ferrite nanoparticles under moderate conditions with good chemical homogeneity and high purity [4]. The objective of the present work is to assess the effect of different organic fuels on the structural and magnetic properties of spinel cadmium ferrite nanoparticles.

2. EXPERIMENTAL

Equimolar quantities of ferric nitrate, cadmium nitrate and citric acid/ urea/ polyethylene glycol were dissolved in minimum amount of distilled water. The mixture was magnetically stirred at 60 °C and ammonium hydroxide was added into the mixture to adjust solution pH to 7.0. After stirring for 6 hours, the solution turned into gel. The gel was dried at 100 °C for 3 hours and finally ground and calcined at different temperatures to get ferrite as final thermolysis product.

3. CHARACTERIZATION TECHNIQUES

Powder X- Ray Diffraction (XRD) patterns have been recorded using a Rigaku Miniflex diffractometer with Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$). The crystallite size was calculated by the Scherrer's formula. Infrared (IR) measurements were recorded using Perkin Elmer – FTIR spectrometer at room temperature using KBr pellet. The morphology studies were carried out using scanning electron microscopy (SEM) Zeiss-Supra 55 model. High resolution transmission electron microscope (Jeol, JEM-2100), operated at 200 kV and equipped with a CCD camera was employed to record the micrographs of the as-synthesized samples. The magnetic properties were measured using a Vibrating Sample Magnetometer (VSM, MicroSense, Model 90).

4. RESULTS AND DISCUSSION

4.1. X-ray diffraction studies:

XRD patterns of Cd ferrites (Fig.1) depict the formation of well crystalline, single phase cadmium ferrite without any impurity peak at different temperature for the three organic fuels. All the XRD peaks have been indexed to Fd $\bar{3}m$ space group with cubic symmetry [5].

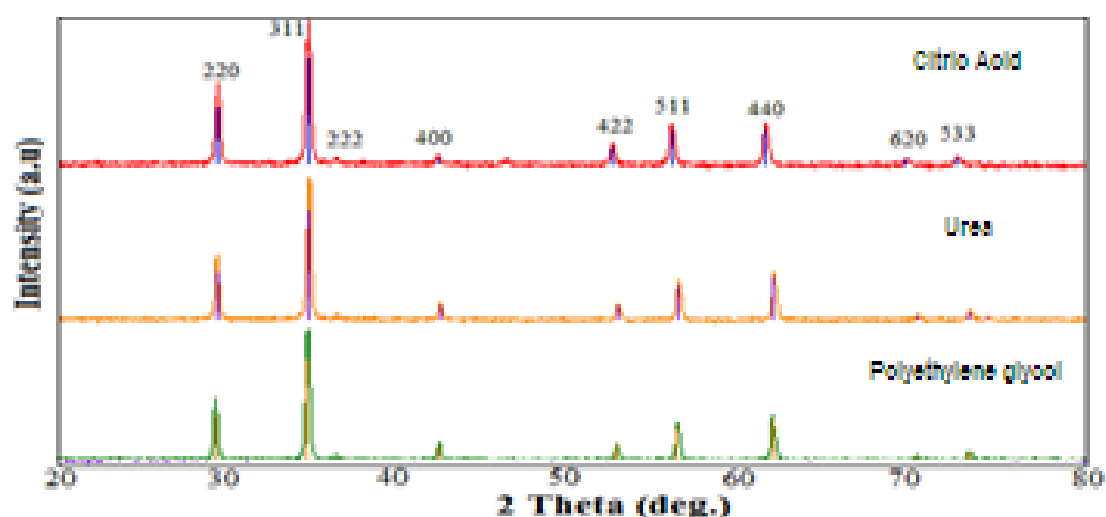


Fig. 1 XRD Patterns for Cd Ferrites prepared using different fuels

Cd ferrite particles were obtained at 500 °C, 600 °C and 650 °C with citric acid, urea and polyethylene glycol respectively.

Lattice constant 'a' for the prepared samples was calculated by using the most intense (311) XRD peak by the equation:

$$a = d_{hkl} (h^2 + k^2 + l^2)^{1/2}$$

where 'd_{hkl}' is the interplaner distance and (h, k, l) are the Miller indices of the planes.

The observed lattice parameter of 8.696 Å for three for three Cd ferrite samples was close to that reported in the literature [6-9].

4.2. Infrared studies \

On the basis of group theoretical calculations along with space group and point symmetry considerations cubic spinel ferrite exhibit four infrared active absorption bands designated as ν_1 , ν_2 , ν_3 and ν_4 . The first three bands are observed due to tetrahedral and octahedral metal complexes while the fourth one is due to some type of lattice vibrations [10]. These four fundamental absorption bands are due to T₂ and E vibrations of symmetry. Absorption bands ν_1 and ν_2 are assigned to the intrinsic lattice vibrations of E type symmetry, while the rest of the bands (ν_3 and ν_4) also are assigned to lattice vibrations having T type symmetry. All the three ferrite samples show almost similar IR spectra as shown in figure 2.

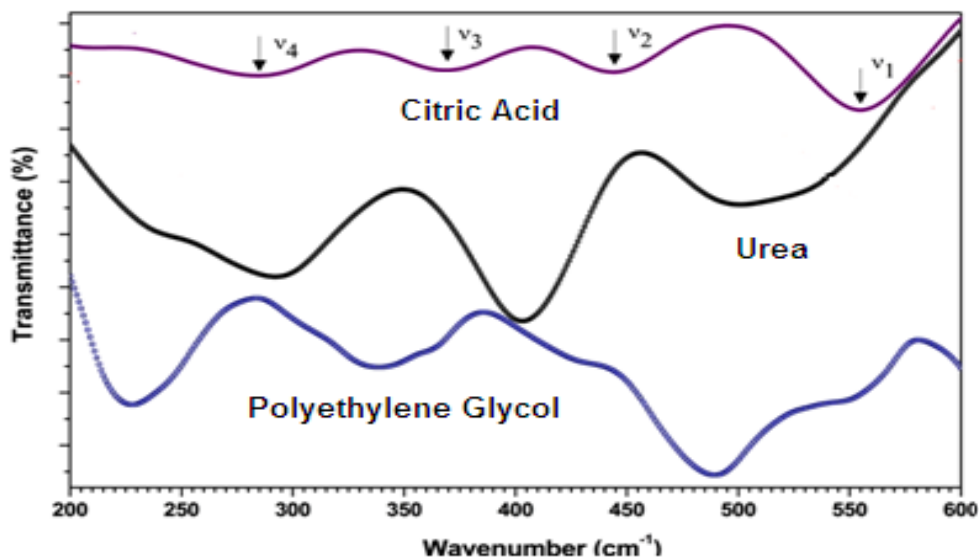


Fig. 2. IR spectra for cd ferrite samples.

4.3. Microstructural Properties

TEM image (Fig. 3) of Cd ferrite samples taken by water dispersion method at high magnification clearly suggest the presence of fine, well dispersed spherical ferrite nanoparticles with average particle diameter of 18-37 nm. With citric acid as the fuel, synthesis temperature has been observed to be at 500 °C and the particle size is about 18 nm. For urea and polyethylene glycol, synthesis temperature and particle size is 600 °C, 27 nm and 650 °C, 37 nm respectively. This increase in particle size at increased temperature resulted due to agglomeration of the ferrite particles.

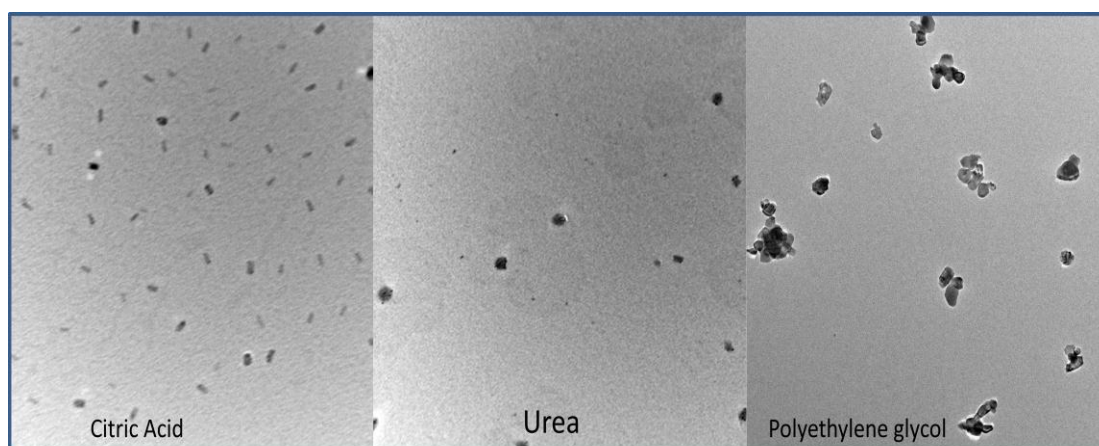


Fig. 3. TEM micrograph for Cd ferrite samples

4.4. Magnetic Study

Room temperature magnetic studies indicate that all the samples displayed normal (S shaped) narrow hysteresis loops (Fig. 4) indicating the soft nature of the ferrites obtained. Narrow hysteresis loops indicated low coercivity suggesting that the prepared samples can be demagnetized easily which is a significant requirement for a superior electromagnet [12]. Cadmium ferrite samples displayed *MS* value of 1.37, 1.28 and 1.03 emu/g for citric acid, urea and polyethylene glycol respectively which are close to the reported ones [11].

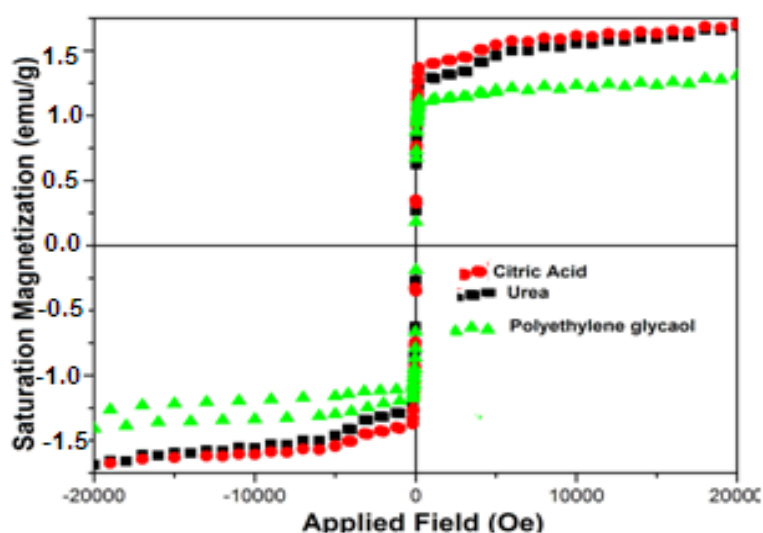


Fig. 4. VSM graph showing variation of saturation magnetization with applied field

5. CONCLUSIONS

A comparative study on the formation of cd ferrite samples with the help of different fuels viz. citric acid, urea, polyethylene glycol indicates the formation of monophasic, crystalline powdered, magnetic materials at temperature conditions of 500, 600 and 650 °C respectively. Various spectroscopic studies employed for the characterization of these three samples obtained differ in size as well as magnetic parameters revealing that properties of ferrite materials are strongly influenced by the distribution of metallic ions among crystallographic lattice sites which in turn is sensitive to the synthetic method employed.

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