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SYNTHESIS OF NANO STRONTIUM HEXAFERRITE BY SOLID-STATE THERMAL DECOMPOSITION

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Abstract

$SrFe_{12}O_{19}$ nanoparticles are synthesized via thermal decomposition of homogenized mixture consisting of stoichiometric amounts of $Sr(NO_3)_2$ and $Fe_2(C_2O_4)_3$. Thermogram of the precursor powder showed a steep weight loss between 50-250^oC followed by a slow weight loss from 250-750^oC. XRD pattern of precursor powder heat treated at 850^oC for 4h indicated formation of phase pure $SrFe_{12}O_{19}$. SEM micrographs revealed the particle size of the calcined powder in the range of 100nm.

Keywords: Strontium hexa ferrite; Thermal decomposition; Nanoparticles; Magnetic materials.

Introduction

Ferrites exist in three different types of crystal structures namely spinel (MFe_2O_4, M=Mn, Fe, Co, Ni, Cu and Zn), Hexagonal ($MFe_{12}O_{19}$, M=Sr, Ba, Pb), and Garnet (MFe_5O_{12}, M=Al, Ga, Y, Gd, Fe). Both spinel ferrites and hexagonal ferrites are technologically very useful because of their electronic, magnetic and catalytic properties. Spinel ferrites are used in permanent magnets, telecommunications, microwave devices, magnetic recording, pigments, humidity sensors, high frequency magneto-optical devices, catalysis etc. [1-6]. Hexagonal ferrites are useful in permanent magnets, catalyst immobilization [7] and electromagnetic interference (EMI) shielding [8]. Strontium ferrite $SrFe_{12}O_{19}$ is an important member of hexagonal ferrites because of its high coercitivity, large saturation magnetization, and high Curie temperature arising from magnetic crystalline anisotropy. $SrFe_{12}O_{19}$ crystallises in magneto plumbite structure in which three Fe^{3+} ions located in octahedral sites and two Fe^{3+} ions located in tetrahedral sites are coupled through super exchange interactions via O^{2-} ions [9].

Traditionally, $SrFe_{12}O_{19}$ powders are prepared by solid-state reaction between $SrCO_3$ and $Fe(NO_3)_3$. $9H_2O$ at temperatures higher than 1200^oC [10]. However, this method yields bigger particles and chemical inhomogeneity. To overcome this, several alternate synthetic routes have been developed in terms of co-precipitation [11], molten-salt assisted co-precipitation [12], hydrothermal [13], sol-gel [14], micro-emulsion [15], spray pyrolysis [16], sonochemical [17], microwave [18] and self propagating combustion methods [19] Though chemical methods offer some advantages over solid-state synthesis in terms of good control over stoichiometry, lower processing temperatures, and better homegeniety, these methods also suffer from some limitations especially in the synthesis of multicomponent oxides. For instance, in the synthesis of $SrFe_{12}O_{19}$ by co-precipitation, high solubility of $Sr(OH)_2$ is a major problem. Likewise in micro emulsion method, though the difficulty in stoichiometric control is reduced, the required calcination temperatures are above 900^oC. Synthesis and applications of hexaferrites have been summarized in a recent review [20]. The present paper describes a facile synthesis for $SrFe_{12}O_{19}$ nanoparticles via solid-state thermal decomposition at temperature lower than those for some of the chemical methods.

Experimental

As purchased samples of A.R. grade $Sr(NO_3)_2$ and $Fe_2(C_2O_4)_3$ are used as starting materials. Stoichiometric quantities of $Sr(NO_3)_2$ and $Fe_2(C_2O_4)_3$ are mixed by grinding for 1 hr in ethanol to yield a homogenized solid precursor which was subsequently heat treated at different temperatures. The resultant powders are characterized for phase identification. Phase identification of calcined precursor samples is done by X-ray diffraction technique using Ni filtered Cu-Kα radiation and a scan rate of 4^o/min in the 2θ range 20-60. Thermal analysis is performed with TG unit using 10^o/min heating rate. Micro structural characterization is performed with a SEM.

Results and Discussion

Thermal behavior of precursor mixture powder in the temperature range of 50-850^{o}C is shown in Fig. 1. From the thermo gravimetric (TG) curve it can be seen that there is a steep weight loss from 50-250^{o}C followed by a slow weight loss up to 750^{o}C. There is no plateau anywhere from 50-850^{o}C which indicates that there is no stable intermediate compound in the entire temperature region of study. No considerable weight loss was observed above 750^{o}C which suggests that thermal decomposition reactions are over by 750^{o}C. The total weight loss observed is about 61% while the theoretical weight loss calculated from the following reaction comes to 57%.

$$Sr(NO_3)_2+6Fe_2(C_2O_4)_3 \rightarrow SrFe_{12}O_{19}+18CO_2+18CO+2NO_2+1/2O_2$$

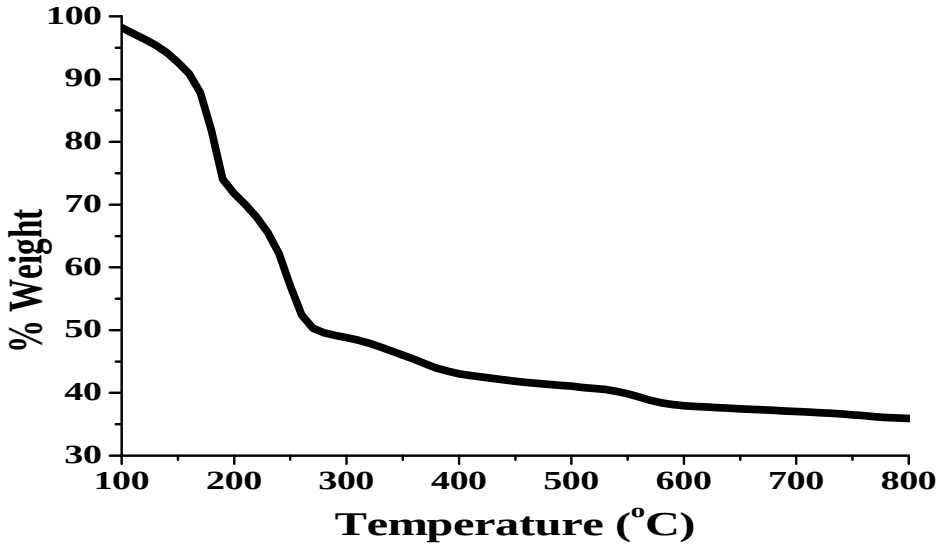

Figure 1. Thermogravimetric curve of homogeneous mixture consisting of stoichiometric amounts of $Sr(NO_3)_2$ and $Fe_2(C_2O_4)_3$

The discrepancy in weight loss may be ascribed to adsorbed water by the sample and remnant ethanol used in grinding. In the sol-gel synthesis of $SrFe_{12}O_{19}$ from $Sr(NO_3)_2$and $Fe(NO_3)_3$. $9H_2O$ the following reactions were proposed to occur:

Precursor $\rightarrow Fe_2O_3+SrO$
$SrO+Fe_2O_3 \rightarrow SrFe_2O_4$
$SrFe_2O_4+5Fe_2O_3 \rightarrow SrFe_{12}O_{19}$

In the TG curve shown in Fig 1, there is no evidence for the formation of either $SrFe_2O_4$ or $SrCO_3$. Hence, in order to check whether the final residue corresponds to a bi-phasic mixture consisting of SrO & Fe_2O_3 or a mono phasic $SrFe_{12}O_{19}$, the precursor powder is heat treated at 700, 850 and 1000^{o}C for 4 hr and their XRD patterns are recorded. From the XRD patterns shown in Fig 2, it can be seen that all the observed peaks in powder calcined at 700^{o}C correspond to formation of $SrFe_{12}O_{19}$. The intensity of peaks increases with increase in calcination temperature from 700 to 1000^{o}C. All the observed peaks in the XRD pattern are in agreement with those reported in JCPDS file No 84-1531 for $SrFe_{12}O_{19}$.

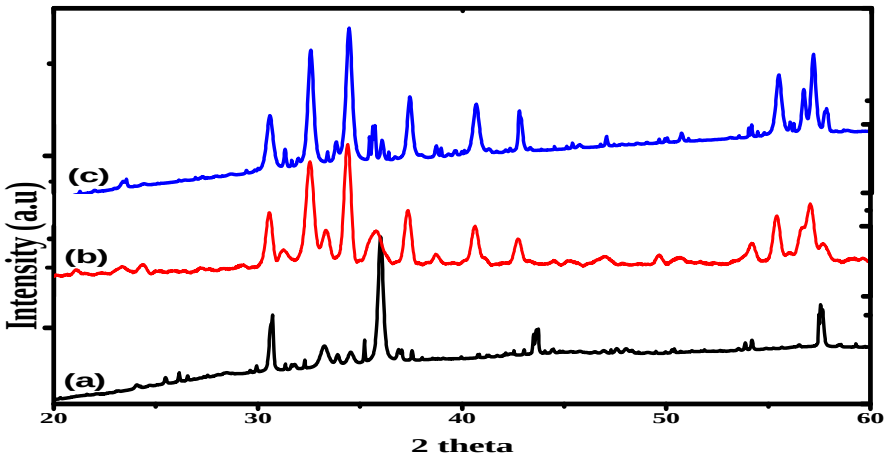

Figure 2. X-ray diffraction pattern of homogeneous precursor powder heat treated at (a) 700°C (b) 850°C and (c) 1000°C

The calculated lattice parameters correspond to a = 5.86247 and c = 23.1162 which agree well with reported values of a = 5.8844 and c = 23.0499. Absence of other peaks characteristic of Fe_2O_3 indicates that formation of phase pure mono phasic $SrFe_{12}O_{19}$ got initiated at temperature as low as 700°C. Micro structural investigation of precursor powder heat treated at 850°C shown in Figure 3 revealed particle size around 100 nm with a characteristic texture of cubelets, some coaleasing into rectangular particles shown in the inset.

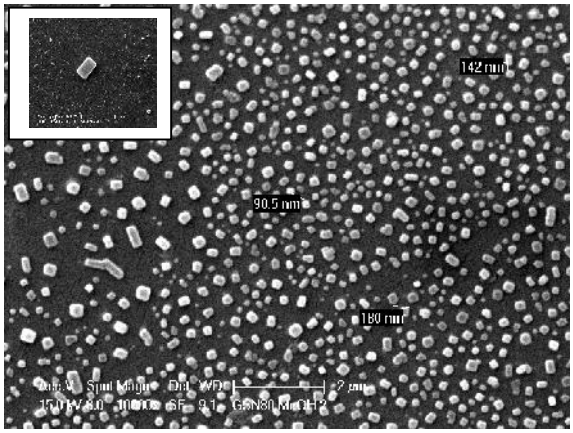

Figure 3. Scanning electron micrograph of homogeneous precursor powder heat treated at 850°C for 4h

The above results confirm that $SrFe_{12}O_{19}$ nano particles can be successfully synthesized by solid-state thermal decomposition method.

Conclusions

Nano particles of $SrFe_{12}O_{19}$ have been successfully synthesized via solid-state thermal decomposition of homogeneous mixture consisting of stoichiometric amounts of $Sr(NO_3)_2$ and $Fe_2(C_2O_4)_3$ as precursors. Thermogravimetric studies indicated no formation of an intermediate compound or other compounds such as $SrCO_3$ or $SrFe_2O_4$. XRD patterns of precursor powder calcined at 700, 850 and 1000°C are indicative of formation of phase pure $SrFe_{12}O_{19}$. SEM micrographs revealed particle size in the range of 100 nm for sample calcined at 850°C.

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