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Formation and characterization of magnetite nanoparticles prepared by the co-precipitation method

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ABSTRACT

Iron oxide powders have been synthesized by co-precipitation method at 700^0C (2h), using X-ray diffraction (XRD), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) have been employed to characterize the magnetite nanoparticles. The results indicate that the formation and stability of magnetite nanoparticles are strongly affected by the precursors. The average diameter of iron nano- particles can be controlled by the iron concentration and thermally treatment.

KEYWORDS

Co-precipitation;
Thermal treatment;
Magnetite;
Hematite;
Nanocrystallites.

INTRODUCTION

Nano binary oxide system with controllable size and properties has applications in miniaturized optical devices, catalysts photonics, advanced high temperature superconductors/ceramics and integrated optics. Magnetic nanoparticles are gaining increasing attention in biomedical applications[1-18]. Iron nano-particles can be prepared by a variety of methods. The problem is that the traditional methods of synthesis from material science are not able to produce uniform and reproducible particles of nanometer size. On the other hand the magnetic particles having nanometer dimensions have the tendency to agglomerate[1]. The co-precipitation method has revealed the formation of the Fe_2O_3 nanoparticles in organic polymers or in silica matrixes[2-4].

It is well known that the structure and composition of nano oxides formed by co-precipitation method depend on the preparation condition, the nature of the precursors, the ion source and pH. All the previous studies have shown that the final product of the decomposition is α-Fe_2O_3, whose properties depend of the temperature of annealing[7], treatment condition[8,9] and the crystallinity of the initial material[10].

The present paper presents the preparation of some Fe_2O_3 nano-particles obtained by the co-precipitation method, recently proposed for the synthesis of Nd_2O_3-SiO_2 systems[19]. The size and morphology of the par-

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ticles were observed by scanning electron microscopy (SEM). The interaction of iron oxide has been investigated by IR-spectroscopy.

EXPERIMENTAL

Magnetite nanoparticles were synthesized by a co-precipitation method. The high purity reagents: Potassium ferri cyanide (K_3 [$Fe(CN)_6$]; Aldrich 99.99%), ferric chloride ($FeCl_3$; Aldrich 99.99%), and deionized water were used. To prepare the sample 100 ml 0.1 M $K_3[Fe(CN)_6]$ aqueous solution was slowly added to 200 ml 0.1 M $FeCl_3$ aqueous solution and the resulting solution was heated up to 40^{0}C. The pH of the resultant solution was 9. To this end, hot solution was allowed to cool at room temperature and diluted to double of its initial volume after cooling. Dark green precipitate, so obtained, was filtered, washed many times with deionized water and acetone and finally allowed to dry in air. The samples were ground to very fine powder. The powder sample calcined in muffle furnace (KSL 1600X, MTI) in air at different heating rates i.e. from room temperature up to 700^{0}C (2h) at 4^{0}C/h. Complementary methods were used to characterize the structure and phase of heat treated samples. X-ray diffraction pattern of samples were carried out by a Philips X-ray diffractometer PW/1710; with Ni filter, using monochromatic CuKα radiation of wavelength 1.5418A^0 at 50KV and 40mA. Scanning electron microscopy (SEM) of the samples was done with JEOL-JSM-T330-A 35 CF microscope at an accelerating voltage of 20KV. Infrared spectra were collected by using Fourier transform infrared spectrometer (Perkin Elmer 1600) ranging 2000-500 cm^{-1}.

RESULTS AND DISCUSSION

XRD

X-ray diffraction investigations have revealed that the samples heat treated in air at temperatures 700^{0}C show signal of crystalline phase. With this temperature from the amorphous background emerge very broad diffraction lines due to weak crystallinity and/or very small dimension of the iron oxide crystalline particles.

In XRD patterns of samples fired at 700^{0}C (Figure 1) at large angle region (30-50^o) appears a broad maximum due to crystalline character. X-ray diffraction pattern presents well developed diffraction peaks that can be undoubtedly ascribed to hematite (a-Fe2O3), with an average particles size of ~20 nm derived from line broadening using Scherrer formula.

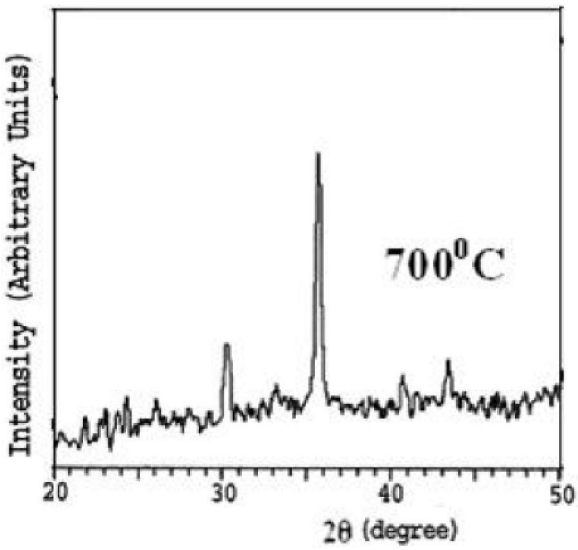

Figure 1 : XRD pattern of the iron oxide powder sample annealed at 700^{0}C (2h).

FTIR

Figure 2 shows IR transmittance spectra (range 2000-500cm^{-1}) of the heat treated sample at temperature 700^{0}C (2h); discrete medium peaks appeared at 695 cm^{-1}. In this low frequency region of FTIR spectra, a medium

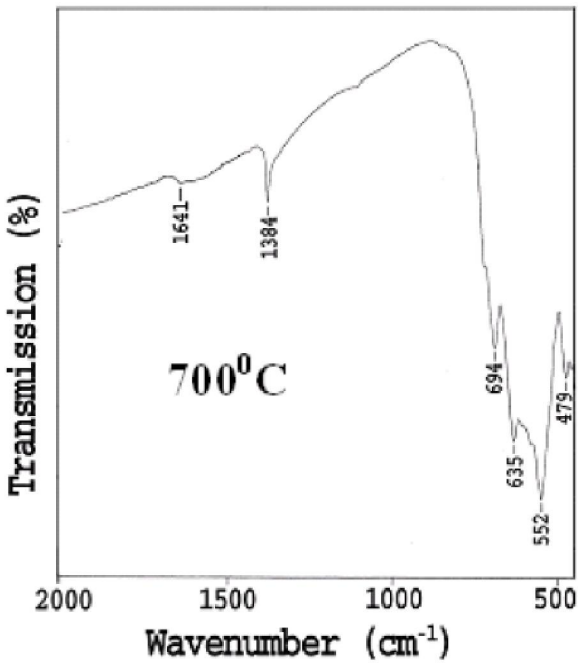

Figure 2 : FTIR spectra of iron oxide at different temperatures: 700^{0}C (2h).

peak appeared at 552 cm^{-1} and this is indicative peak of the Fe-O stretching vibration in iron oxide. Beside, the peak centered about 635 and 694 cm^{-1} may be assigned to α-FeOOH and β-FeOOH phase, respectively. In addition, the peak centered at 1384 cm^{-1} can be ascribed to C-H bending vibration mode. The weak band centered around 1641 cm^{-1} is due to the bending mode of H-O-H adsorbed at the Fe_3O_4 surface. FTIR results also support the XRD data.

SEM

The influence of the iron source on the properties of the prepared nanoparticles is also evidenced in Figure 3, which presents the SEM micrographs of the samples The dark field micrographs indicate the amorphous character of the majority of nanoparticles in the samples treated at temperatures 700 ºC (2h). The white patches in the micrograph reveal the crystallization of the samples. In this image one may also see clearly that particles have lamellar structures of irregular shapes and different size in the range 17-35 nm. Image also exhibits weak aggregation of particles, which was expected due to the technique used for the sample preparation. The weak agglomerate structure indicates development of super paramagnetic structure. The SEM data also supports the XRD data.

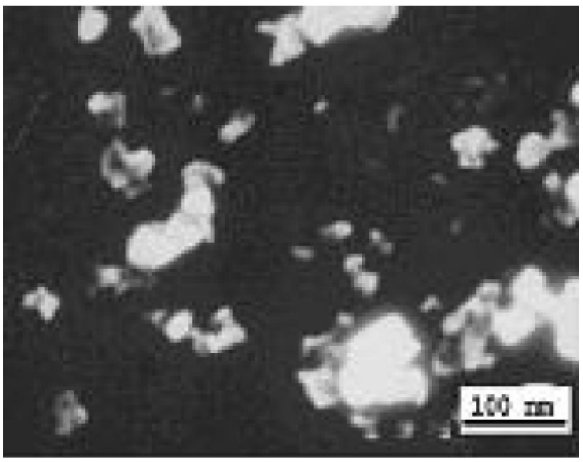

Figure 3 : SEM photograph of iron oxide at 700^{0}C (2h).

CONCLUSIONS

In summary, the co-precipitation method has been used to successfully produce the magnetite nanoparticles. The presence of the dark-field micrographs indicates the amorphous character of the nanoparticles in the samples. The tendency of crystallization of iron oxides increases in the case of the material obtained. We believe that these hydrophilic and biocompatible nanoparticles will have important applications not only in advanced magnetic materials and ferrofluid technology, but also in biomedical fields such as biomolecular separations, targeted drug delivery, cancer diagnosis and treatment, as well as magnetic resonance imaging.

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