Synthesis and Characterization of Fe₃O₄ Nanopowders using Co-precipitation Method

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ABSTRACT

Nanoferrite, magnetite (Fe₃O₄) nanoparticles were synthesized via facile, aqueous co-precipitation method from FeSO₄ and NaOH as iron precursor and reducing agent respectively. Structural studies reveal that pH and precursor molar concentration play an important role in obtaining the Fe₃O₄ nanoparticles. FTIR spectra further confirm the presence of functional group corresponding to Fe₃O₄ in the prepared samples. Such nanostructures powders can be useful for various applications such as EMI shielding, drug delivery and biosensing.

Introduction

Nanoparticles of magnetic metal oxides are very important, especially at nanoscale in biomedical, environment and industrial world today. Nanopartculate iron oxide, especially magnetite (Fe₃O₄) is a kind of excellent magnetic material [1]. Due to its magnetic properties, biocompatibility and cost effectiveness, it is explored for various applications such as EMI shielding, drug delivery, tumour treatment, MRI contrast agent, bio-sensing, gas sensing, etc [2,3]. Fe₃O₄ nanoparticles have been synthesized using various techniques like hydrothermal, sol-gel, co-precipitation, emulsion, reverse-micelle, etc [2, 4]. Among these, co-precipitation is simple, cheap, environmental friendly and scalable nanomaterials synthesis technique. Therefore, it was used in the present work for the aqueous synthesis of Fe₃O₄ nanoparticles at room temperature.

EXPERIMENTAL PROCEDURE:-

Materials

 $FeSO_4.7H_2O$ and NaOH were of analytical grade and used without any further processing for the synthesis of (Fe_3O_4) nanoparticles using aqueous co-precipitation technique.

Experimental work

Three clean beakers of 250ml volume capacity were taken and named as beaker A, B and C for carrying out three different sets of co-precipitation reactions under the experimental conditions given in Table I. In a typical procedure, 80 ml FeSO₄.7H₂O of predetermined molar concentration was dissolved in the beaker. Aqueous NaOH solution of known molar concentration (80 ml), which was separately prepared in another beaker, was added to this solution under constant magnetic stirring. The mixture of these two solutions was stirred for another 30 min at 250rpm. The precipitate was washed by centrifugation 2 times by distilled water and lastly by ethanol to remove un-reacted materials. The residue was dried in oven for 60 minutes at 100 °C to obtain different coloured powders for different reaction conditions.

Sr.no.	Conc. of FeSO ₄ .7H ₂ O	Conc. of NaOH (M)	Sample code
	(M)		
1	0.20	1.00	Sample A
2	0.20	2.00	Sample B
3	0.10	1.00	Sample B

Table I: Molar concentrations of iron precursor and reducing agent and their sample codes

Characterization

The structural properties of the sample were studied using X-ray diffraction (XRD) technique. The information of functional groups present in the samples was obtained using Fourier transform infra-red (FTIR) spectroscopy. Optical properties of the prepared samples were studied using UV-visible spectroscopy.

Result and discussion

XRD patterns of as prepared powder samples are shown in Fig.1. The XRD data clearly confirm the formation of cubic crystalline structure of ferrite (Fe₃O₄) when matched with JCPDS No. 39-1346 for all the samples. For the prepared Fe₃O₄ powder samples, the most intensive peaks at 2θ =35.440 and 62.90 corresponding to (311) and (440) respectively are observed. The samples show broad peaks, indicating the small crystallite size (nanoscale) of the particles. However, in case of sample A, impurity peaks are also observed which may be due to incomplete phase formation at lower pH yet higher molar concentration of FeSO₄.7H₂O. FTIR spectra of Fe₃O₄ powder samples are shown in Figure 2. The spectra shows presence of two peaks at 584 and 442 cm⁻¹ which correspond to normal mode of vibration of tetrahedral cluster and octahedral cluster respectively for Fe₃O₄ [1]. Thus formation of Fe₃O₄ is confirmed by XRD and FTIR techniques.

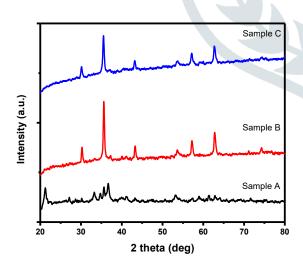


Figure 1: XRD patterns of Fe₃O₄ powder samples

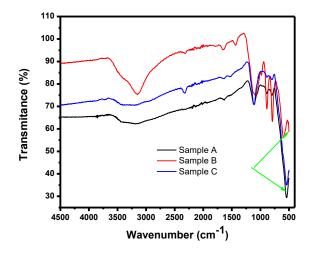


Figure 2: FTIR spectra of Fe₃O₄ powder samples

Conclusions

Nano sized Fe₃O₄ powder has been successfully obtained using convenient, environment friendly, inexpensive and efficient co-precipitation method.

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