

NOTE

Preparation and Characterization of Fe₃O₄ and Fe₃O₄/Bentonite Nanocomposite by Co-precipitation Method

SAEEDAH HASHEMIAN* and HOSSEIN SAFFARI

Department of Chemistry, Scientific Association of Nanotechnology, Islamic Azad University, Yazd Branch, Yazd, Iran

*Corresponding author: Fax: +98 351 6240020; Tel: +98 351 8211391-9(572); E-mail: sa_hashemian@yahoo.com

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The particles of Fe₃O₄ and Fe₃O₄/bentonite were prepared by co-precipitation method. X-Ray diffraction patterns indicated that the magnetic nanoparticles were pure Fe₃O₄ with a spinel structure and the binding of bentonite did not result in the phase change. The FTIR was used for characterization of these nano-ferrites. The TEM image of Fe₃O₄/bentonite was shown the particles diameters were 10 nm.

Key Words: Co-precipitation, Fe₃O₄/bentonite, Nanocomposite.

Spinel ferrites are known to be materials extremely sensitive to the manufacturing process. Composite oxides with spinel structures (AB₂O₄) are important inorganic metalloid materials and are widely used in different fields. They can be distinguished as two basic types, normal and inverse spinels. The formula AB₂O₄, where A and B denote two different sites in the crystalline structure. The A-site is tetrahedral and the B-site is octahedral coordinated by oxygen. In the normal spinel, A-site is occupied by divalent ion M and B-site the trivalent ion M. In an inverse spinel, the formula becomes tetrahedrally coordinated by oxygen and the A, B in side bracket is octahedrally coordinated by oxygen. It was found that such kinds of ferrites are characterized by a spinel structure with high thermodynamic stability, electrical conductivity and high catalytic activity. The catalytic activity of the obtained pure and mixed metal oxides such as CuO-Fe₂O₃ for adsorption of pollutants and dyes was measured¹⁻⁴. FTIR study of Ni, Cu and Zn substituted nano-particles of MgFe₂O₄ was done⁵. Catalytic oxidation of CO and adsorption of Zn, Cu and dyes by spinels were also studied⁶⁻¹⁰.

The objective of this work is to synthesize magnetic ferrite and magnetic ferrite composite by co-precipitation method and characterize their structure and morphological properties.

Ferrous(II) chloride dehydrate, ferric(III) chloride were obtained from Merck. All of other compounds were analytical grade.

Fe₃O₄ and Fe₃O₄/bentonite magnetic composites were prepared using a co-precipitation method. The bentonite was added into a 400 mL solution containing ferrous(II) chloride

(0.02 mol) and ferric(III) chloride (0.04 mol) at room temperature. The amount of bentonite was adjusted to obtain Fe₃O₄/bentonite mass ratio of 1:10 under vigorous magnetic-stirring, slowly raised the pH by adding NaOH (pH-5) solution to around pH-10 and stirring was continued for 0.5 h and the stopped stirring. The suspension was heated to 95-110 °C for 2 h. After cooling, the prepared magnetic composite was repeatedly washed with distilled water. By a simple magnetic procedure, the obtained materials were separated from water and dried in an oven at 110 °C. The crystalline structure of Fe₃O₄ in the composite was determined *via* the X-ray powder diffraction method using an Endeavor D4 with CuK_α radiation. pH measurement was done with a Horiba pH meter (M13, Japan). Infrared spectrum is recorded using FTIR instrument (Burrker, Tensor T27) for all samples in KBr medium between the 4000 and 400 cm⁻¹.

Characterization of adsorbent (composites): The composites were prepared by the precipitation of Fe₃O₄ onto bentonite surface. The magnetism test with a permanent magnet (2300 G) showed that the material was magnetic and completely attracted to the magnet. Fig. 1 shows the XRD diffraction patterns for bentonite, Fe₃O₄, Fe₃O₄/bentonite nano particles. It indicated that Fe₃O₄ is the dominant phase in both samples of Fe₃O₄, Fe₃O₄/bentonite. The characteristic peaks for Fe₃O₄ were observed for both samples (these peaks are consistent with the data base in JCPD file) and reveal that the resultant nanoparticles were pure Fe₃O₄ with a spinel structure. It is also explained that the mixing process did not result in the phase change of Fe₃O₄.

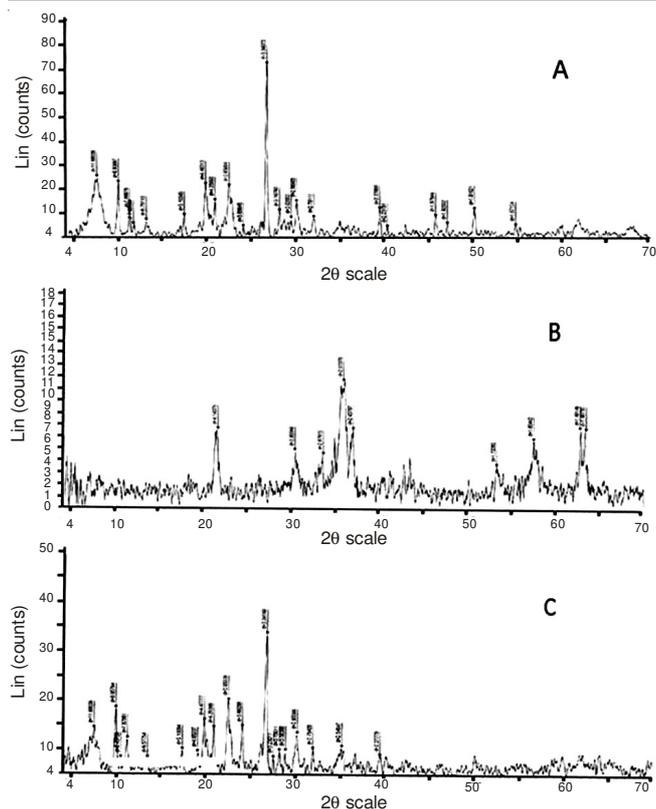


Fig. 1. Powder XRD patterns for the (a) bentonite, (b) Fe_3O_4 and (c) Fe_3O_4 /bentonite composite

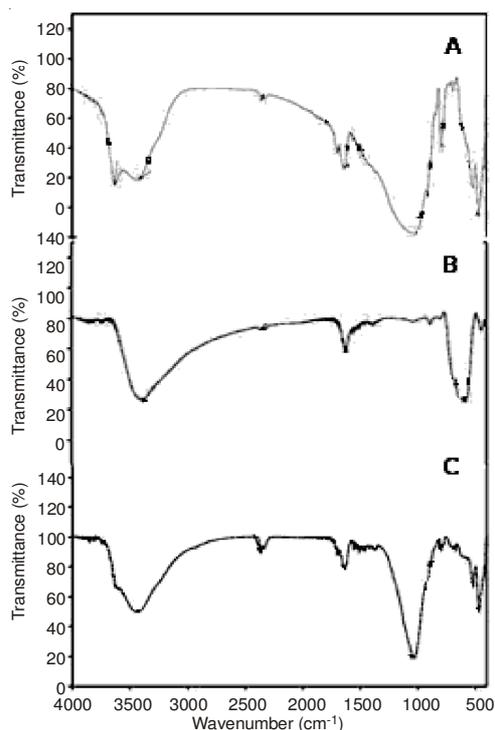


Fig. 2. FTIR spectra of (a) bentonite, (b) Fe_3O_4 and (c) Fe_3O_4 /bentonite composite

The effect of dependence of normal modes and their frequency data for the respective sites are analyzed. In the FTIR spectrum of bentonite, the peaks at 3632 and 3426 cm^{-1} are O-H stretching and H-O-H hydrogen binding water. The peak of 1638 cm^{-1} is due to H-O-H deformation. The peak of 1021 cm^{-1} is assigned for Si-O-Si, sharp peak of 783 cm^{-1} is due to Si-O stretching of quartz and silica, peak of 514 cm^{-1} Al-O-Si deformation and the peak of 467 cm^{-1} Si-O-Si deformation. In the FTIR of Fe_3O_4 , the peak at 581 cm^{-1} relates to Fe-O group. In the FTIR spectrum of Fe_3O_4 /bentonite composite, the peak 467 cm^{-1} shifted to 471 and 514-518 cm^{-1} and peak of 783 shift to 795. Fig. 2 shows FTIR spectra of (a) bentonite, (b) Fe_3O_4 and (c) Fe_3O_4 /bentonite composite.

The typical TEM micrograph of the Fe_3O_4 /bentonite nanoparticles is shown in Fig. 3. It was clear the nanoparticles had a mean diameter of 10 nm. This reveals that the binding process did not significantly result in the agglomeration. This could be attributed to the reaction occurring only on the particle surface.

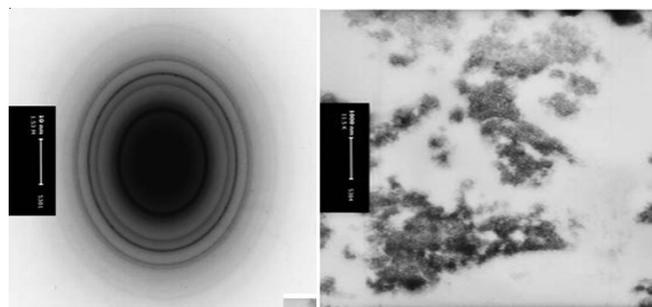


Fig. 3. TEM image of Fe_3O_4 /bentonite nanocomposite

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