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Synthesis of cubic CuFe₂O₄ nanoparticles by microwave-hydrothermal method and their magnetic properties



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ABSTRACT

Copper ferrite ($CuFe_2O_4$) nanoparticles were prepared in alkaline solutions containing $Cu(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ with the pH of 6–12 by microwave-hydrothermal method. The as-prepared products characterized by XRD were specified as cubic $CuFe_2O_4$ phase. Average sizes of the $CuFe_2O_4$ nanoparticles calculated by Scherrer formula are in the same range as those obtained by TEM analysis. FTIR shows main transmittance band at 585 cm $^{-1}$, corresponding to stretching mode of tetrahedral complexes. Saturation magnetization, remanence and coercivity were increased with increasing in the particle-size and crystalline degree of the nanoparticles.

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1. Introduction

Copper ferrite ($CuFe_2O_4$) has been widely investigated its properties for using as sensors, magnetic materials, anodes, semiconductors and catalysts [1–4]. $CuFe_2O_4$ has two structures (tetragonal and cubic), controlled by preparation conditions [4,5]. Microwave-hydrothermal (MH) method has been used and developed to prepare nanosized compounds because of its fast heating rate, extremely rapid in crystallization of materials and save energy consumption comparing to conventional-hydrothermal (CH) method [6,7]. The objective research was to prepare cubic $CuFe_2O_4$ nanoparticles by MH method. Phase, morphology and magnetic properties were further investigated.

2. Experiment

Typically, $0.005 \text{ mol } \text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 0.010 mol Fe $(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were dissolved together in 40 ml deionized water to

form solution under magnetic stirring until the reactants were completely dissolved. Each of the solutions was adjusted the pH to 6, 8, 10 and 12 by adding of 3 M KOH dropwise and separately loaded into a 100 ml TFM fluoropolymer lining vessel. The vessels were tightly closed and transferred in a CEM Mars-5 microwave oven. The system was heated by 300 W microwave from room temperature (T_R) to 150 °C for 20 min, held at this temperature for 60 min and cooled to T_R . All products were filtered, washed with deionized water and ethanol, and dried at 80 °C for 24 h.

3. Results and discussion

XRD patterns (Fig. 1a) of CuFe₂O₄ prepared in the solutions with different pH values by MH method show peaks at 2θ of 30.15°, 35.54°, 37.20°, 43.19° and 57.06° which were indexed to the (220), (311), (222), (400) and (511) planes of cubic CuFe₂O₄ (JCPDS database no 25-0283) [8]. Impurities such as CuO, Cu₂O, Fe₂O₃ and others were not detected in these samples. According to the previous report, pure CuFe₂O₄ was prepared by CH method at 160 °C for 30 h [9]. The MH is a one-step method, which takes shorter reaction time at lower temperature and consumes less energy than the CH one. Upon increasing in the basicity of the solutions (pH: $6 \rightarrow 12$), intensities of the diffraction peaks became strengthened and their widths were narrowed, implying that

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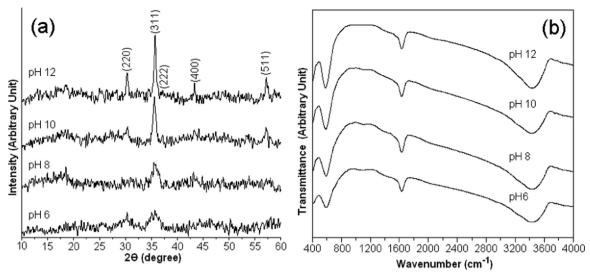


Fig. 1. (a) XRD patterns and (b) FTIR transmittance of CuFe₂O₄ prepared in different solutions.

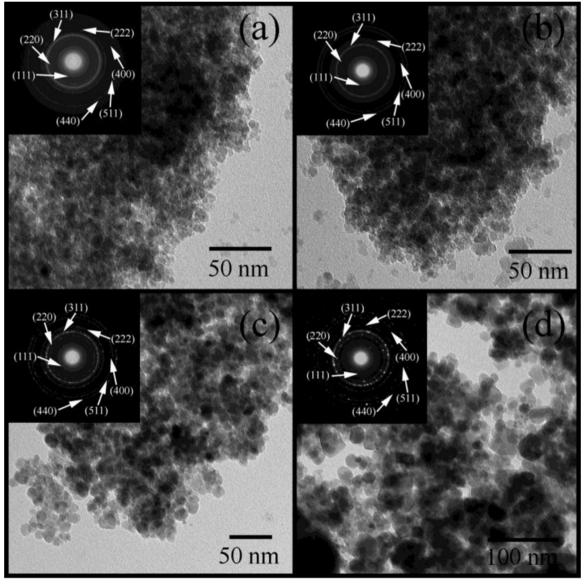


Fig. 2. TEM images and SAED patterns of CuFe₂O₄ prepared in different solutions with the pH of (a-d) 6, 8, 10 and 12, respectively.

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