SYNTHESIS OF UNIFORM CUBE SHAPE $CuFe_2O_4$ NANOPARTICLES BY A HYDROTHERMAL METHOD

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1. INTRODUCTION

Magnetic nanoparticles (NPs) have been of great interest because of its attractive features and wide range of extensive applications in catalyst, adsorption, and as a supercapacitor electrode [1-3]. The size and shape of NPs determine their physical and chemical features, which may funtion as a foundation for the development of new product [4, 5]. As a conventional magnetic material, magnetite Fe_3O_4 and ferrites MFe_2O_4 have yielded a great deal of paper featuring mumerous techniques and nanoparticle morphologies [6, 7].

In a previous report, solvothermal strategy has been widely used to synthesize many kinds of NPs with uniform size and shape, including monodisperse nanocrystals and microspheres MFe_2O_4. Controling the shape of NPs is also an equally improtant aspect of nano synthesis. However, the challenge to synthetically control the morphology of MFe_2O_4 nanostructures with a simple method still remain up to date [8, 9]. Compared with other ferrites, $CuFe_2O_4$ NPs has attracted more attention due to its property and application in catalysis for it is inexpensive and environmental friendly [10, 11]. Additional, $CuFe_2O_4$ NPs can be recovered conveniently after the reaction by a magnet [12-14].

In this study, monodisperse NPs cube shape $CuFe_2O_4$ were successfully synthesized through a hydrothermal method. The $CuFe_2O_4$ NPs has a superparamagnetic and an uniform cube shape structure.

2. EXPERIMENTAL

2.1. Material

Iron(II) sulfate heptahydrate ($FeSO_4.7H_2O$), oleic acid (OA), ethanol (EtOH), sodium hydroxide (NaOH), copper sulphate pentahydrate ($CuSO_4.5H_2O$) were purchased from Aladdin Chemical Co., Ltd. All the reagents were of analytical grade and used without further purification, and solution were prepared using de-ionized water.

2.2. Synthesis of cube shape $CuFe_2O_4$ NPs

In a typical synthesis, 1.5 g NaOH, 15 mL H_2O, 9 ml ethanol, and 15 mL oleic acid (OA) were mixed together to form an even solution. After stirring for 30 min, an aqueous solution of 2 mmol $FeSO_4.7H_2O$ (0.56 g) and 1 mmol $CuSO_4.5H_2O$ (0.25 g) (in 21 mL de-inozed water) was the added. After further stirring for 30 min, the solution was transferred into an autoclave and kept at 160^{o}C, 180^{o}C, 200^{o}C for 10h, respectively. The system was then allowed to cool to room temperature. The $CuFe_2O_4$ products were isolated by strong magnetic suction, and washed with ethanol and deionized water several times [3].

2.3. Characterization

Powder X-ray diffraction (XRD) spectra were obtained by a Rigaku D/max-2400 diffractometer using Cu-Kα radiation in the 2θ range of 10-90°. Transmission electron microscopy (TEM) images were obtained on a Tecnai G2 F30, FEI, USA. SEM images was collected on a Hitachi S-4800 field emission scanning electron microscope equipped with a Horiba EMAX energy-dispersive X-ray analyser. Magnetic measurements of $CuFe_2O_4$ NPs were investigated with a quantum design vibrating sample magnetometer (VSM) at room temperature in an applied magnetic field sweeping from -15 to 15 kOe.

3. RESULTS AND DISCUSSION

The morphologies and structural of the synthesized $CuFe_2O_4$ NPs were analyzed by SEM. As is illustrated in Fig. 1 (a,b and c) with a uniform cube shape, resulting from a minimized surface energy.

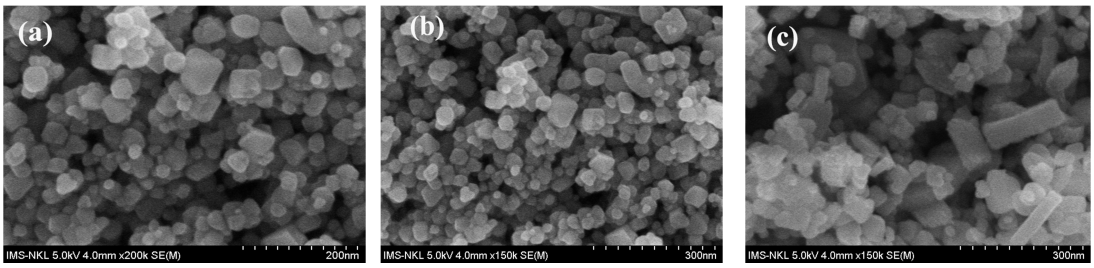

Fig. 1. SEM image of the $CuFe_2O_4$ NPs formed at different temperatures; (a) 160°C, (b) 180°C and (c) 200°C

We can draw from Fig. 1 that the size of $CuFe_2O_4$ NPs increased with the increase of reaction temperature.

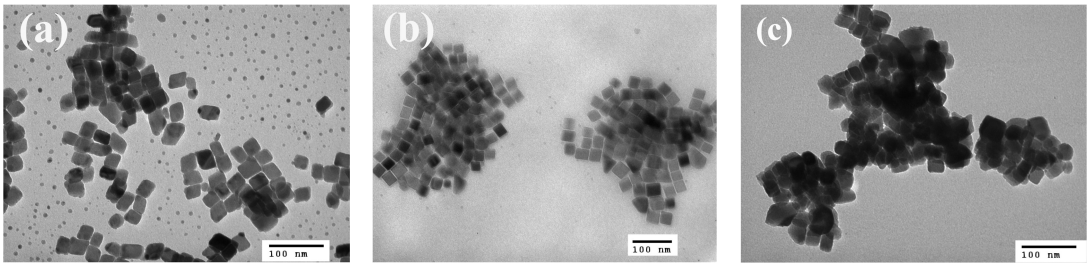

Fig. 2. TEM image of the $CuFe_2O_4$ NPs formed at different temperatures; (a) 160°C, (b) 180°C and (c) 200°C

TEM image (Fig. 2) confirms the $CuFe_2O_4$ NPs shape is cube structure. The particles were well dispersed with a mean particle size of about 50 nm.

The XRD patterns of the $CuFe_2O_4$ NPs is shown in Fig. 3. The XRD pattern of the $CuFe_2O_4$ NPs shows the characteristic peaks of magnetite NPs. The sharp and strong peaks confirm that the products are well crystallized. The $CuFe_2O_4$ NPs show five characteristic diffraction peaks at 2 theta = 30.3°, 35.6°, 43.2°, 57.2° and 63.0° corresponding to (220), (311), (400), (511), and (440), respectively [15].

From the SEM, TEM and XRD, we can draw conclusions. The reaction temperature at 180°C is the best condition for synthesized unifrom cube shape $CuFe_2O_4$ NPs.

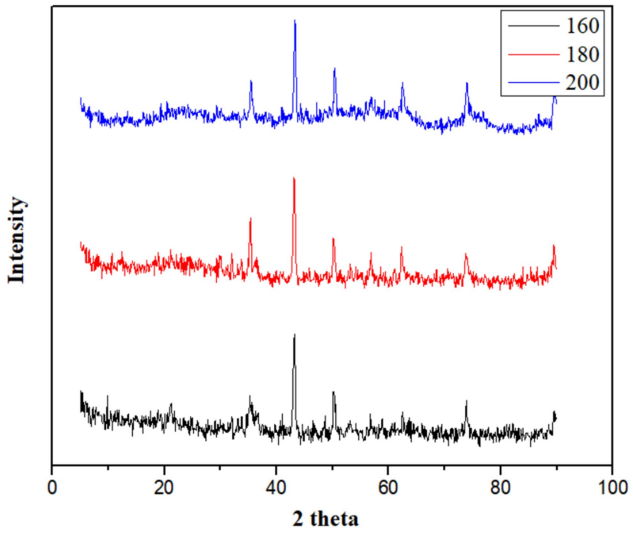

Fig. 3. XRD of $CuFe_2O_4$ NPs formed at different temperatures; (a) 160°C, (b) 180°C and (c) 200°C

Fig. 4 shows the FT-IR spectra of $CuFe_2O_4$ NPs. The IR spectra show main absorption bands at ~580 cm^{-1}, corresponding to the the metal oxygen stretching vibrations of octahedral and tetrahedral ions [15]. The absorption broad band at ~3400 cm^{-1} represents the stretching mode of H_2O molecules and OH groups. The band around 1600 cm^{-1} is corresponds to the bending mode of H_2O molecules.

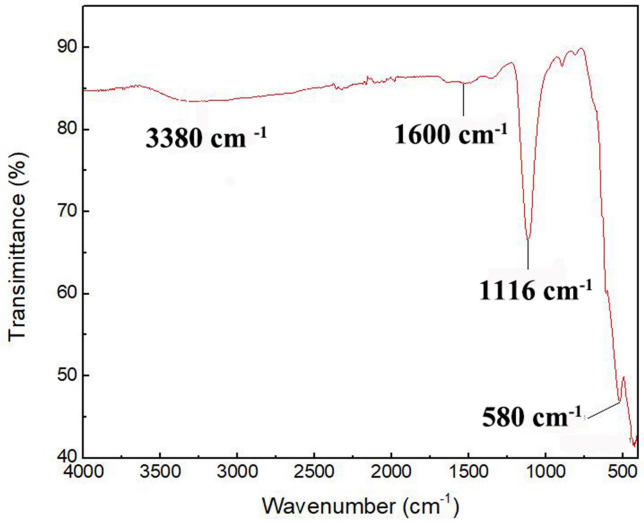

Fig. 4. FT-IR spectra of $CuFe_2O_4$ NPs with reaction temperatures 180°C

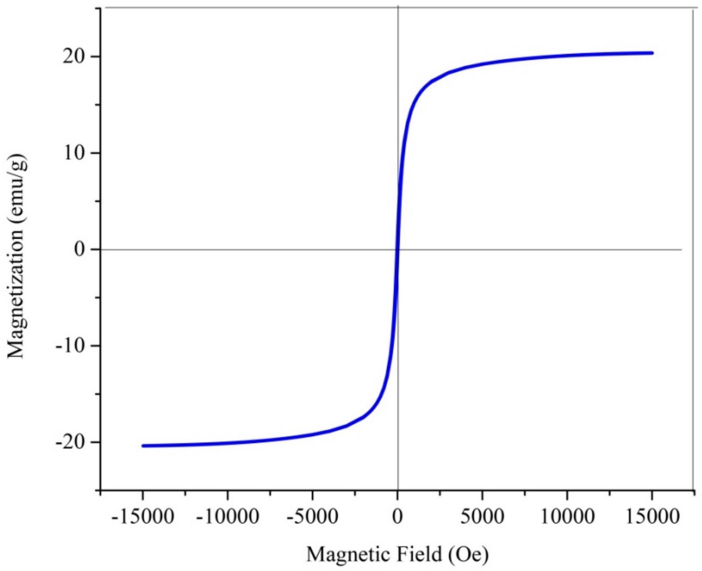

Fig. 5. Room temperature magnetization curves of the $CuFe_2O_4$ NPs with reaction temperatures 180°C

The magnetic measurements were carried out by VSM at room temperature. The magnetization curves measured for $CuFe_2O_4$ is shown in Fig. 5. The magnetic saturation values of $CuFe_2O_4$ is 20.5 emu/g. The abovementioned results indicated an easy and efficient way to separate and recycle the $CuFe_2O_4$ from the solution by an external magnetic field.

4. CONCLUSION

In summary, $CuFe_2O_4$ NPs which features with superparamagnetic, and cube shape structure was synthesized by a hydrothermal method. It can also be valuable in catalyst, medicine, and as supercapacitor electrode, and in nano composite materials.

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SUMMARY

In this study, $CuFe_2O_4$ nanoparticles (NPs) which features with superparamagnetic, and uniform cube shape structure was synthesized by a hydrothermal method. The prepared samples were characterized by scanning electron microscope (SEM), transmission electron microscopy (TEM), vibrating sample magnetometer (VSM), X-ray powder diffraction (XRD). The $CuFe_2O_4$ NPs were well dispersed with a mean particle size of about 50 nm. The $CuFe_2O_4$ NPs is extremely useful for support catalyst in heterogeneous catalysis applications and adsorption.

Keywords: *Cube shape $CuFe_2O_4$, superparamagnetic, nanoparticles.*

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