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Synthesis of $NiFe_2O_4$ Powders Well Defined in Size and Morphologies

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Abstract: The uniform $NiFe_2O_4$ powders with different particle size and morphologies (octahedral, cubic and spherical) have been prepared from different precursors *via* hydrothermal process. The nanocrystallines derived from precursor B in the weak alkali solution ($pH \leqslant 10$) are superparamagnetic.

Keywords: Nickel ferrite, hydrothermal method, superparamagnetism.

In recent years, the superparamagnetic nanocrystallites have attracted more attention for their various applications including ferrofluid technology, magnetically guided site-specific drug delivery, microwave devices, contrast enhancement of magnetic resonance imaging (MRI) and *etc*[1]. Although $NiFe_2O_4$ powders have been successfully synthesized by other methods before[2,3], but the synthetic methods for preparing high crystallized and uniform $NiFe_2O_4$ particles with a narrow size distribution are still lacking. In this work, we successfully prepare the high quality superparamagnetic $NiFe_2O_4$ particles well defined in size and morphology through the hydrothermal process.

Experimental

Using α-FeOOH powders and amorphous $Ni(OH)_2$ (precursor A) or the mixed metal hydroxides of Fe^{3+} and Ni^{2+} (precursor B) as starting materials, the above precursor A or B was dispersed in a NaOH solution and stirred for 30 min. Then the as-prepared suspension was poured into a teflon-lined autoclave and hydrothermally heated at a designed temperature for a planned period. After that the autoclave was cooled to room temperature, the product was collected and then dried at ambient temperature before being characterized.

Results and Discussion

The SEM and TEM photographs of the $NiFe_2O_4$ particles derived from precursor A and B respectively are shown in **Figure 1**. After hydrothermal treatment in alkali medium, precursor A transforms into uniform octahedral microcrystals (*ca*. ~1.23 μm), and

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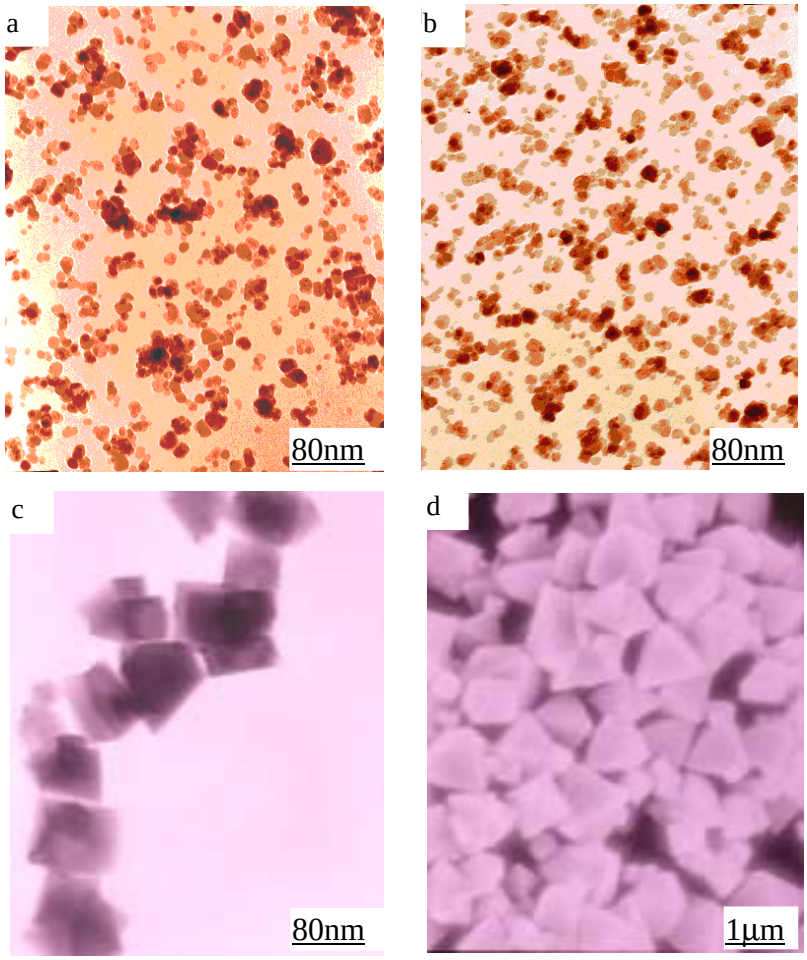
precursor B precipitated in the form of spherical (9~18 nm) or cubic (82~96 nm) nanoparticles, which dependes on the alkali concentration.

Figure 1 The SEM and TEM photographs of the $NiFe_2O_4$ particles obtained at 200℃



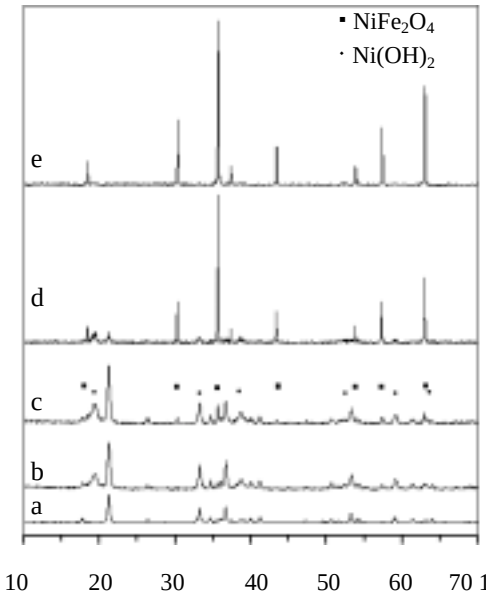
*alkali concentration: a- pH=7, b- pH=10, c- pH=13 from precursor B and d- 4.0 mol/L from precursor A.

The XRD patterns of precursor A and its crystallization behavior while the reaction was held at 200°C for 6 h are shown in **Figure 2**. It is found that the crystallization of $NiFe_2O_4$ was greatly accelerated with the increasing of the alkali concentration. For precursor B, $NiFe_2O_4$ can be obtained even in the neutral medium. Increasing pH value of the medium, the large particles will form, which are demonstrated by the narrowing phenomenon of the corresponding X-ray diffraction peaks (see **Figure 3**).

The magnetic characterization of the as-prepared $NiFe_2O_4$ powders manifestes that the spherical nanoparticles derived from precursor B in weak alkali solution (pH≤10) are

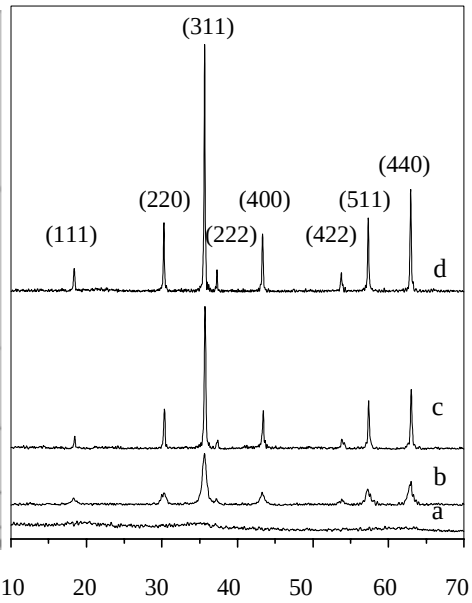
superparamagnetic and the results are fitted with the Langevin equation. These results confirm that the as-prepared $NiFe_2O_4$ powders possess the superparamagnetic properties[4]. Compared with the $NiFe_2O_4$ powders synthesized by other methods[3,5], the resulting powders have higher saturation magnetization moment (M_s) values (see **Table 1**), which might be due to their high crystallization and uniform morphologies. All these properties are interesting for their further applications.

Figure 2 XRD patterns of precursor A and the as-prepared $NiFe_2O_4$ powders derived from precursor A



*precursor A (a), in 0.5 (b), 0.7 (c), 1.0 (d) and 2.0 mol/L• (e) alkali solution.

Figure 3 XRD patterns of precursor B and the as-prepared $NiFe_2O_4$ powders derived from precursor B



*precursor B (a), in pH=7 (b), pH=10 (c), and pH=13 (d) alkali solution.

Table 1 The magnetic parameters of as-prepared $NiFe_2O_4$ powders with different particle size

Sample	Particle size (nm)		M_s value	Corcivity
	XRD*	TEM	(emu g^{-1})	(KAm^{-1})
a	14.2	9~18	48.07	——
b	14.6	10~20	48.72	——
c	89.2	82~96	54.02	54.1
d	——	1.23μm	54.15	54.8

* Calculated from the FWHM of the d_{311} peak using the Scherrer equation.

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