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The dependence of Ni-Fe bixide composites nanoparticles on the FeCl_2 solution used

Yueqiang Lin, Jian Li*, Lihua Lin, Xiaodong Liu, Longlong Chen and Jun Fu

Abstract

Background: Ni_2O_3 - $\gamma\text{-Fe}_2\text{O}_3$ composite nanoparticles coated with a layer of $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$ can be prepared by co-precipitation and processing in FeCl_2 solution. Using vibrating sample magnetometer (VSM), X-ray diffraction (XRD), transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS) diffraction techniques, the dependence of the preparation on the concentration of the FeCl_2 treatment solution is revealed.

Results: The magnetization of the as-prepared products varied non-monotonically as the FeCl_2 concentration increased from 0.020 M to 1.000 M. The Experimental results show that for the composite nanoparticles, the size of the $\gamma\text{-Fe}_2\text{O}_3$ phase is constant at about 8 nm, the Ni_2O_3 phase decreased and the $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$ phase increased with increasing concentration of FeCl_2 solution. The magnetization of the as-prepared products mainly results from the $\gamma\text{-Fe}_2\text{O}_3$ core, and the competition between the reduction of the Ni_2O_3 phase with the increase of the $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$ phase resulted in the apparent magnetization varying non-monotonically.

Conclusions: When the concentration of FeCl_2 treatment solution did not exceed 0.100 M, the products are spherical nanoparticles of size about 11 nm; their magnetization increased monotonically with increasing the concentration of FeCl_2 solution due to the decreasing proportion of Ni_2O_3 phase.

Keywords: Composite, Nanoparticles, FeCl_2 solution, Concentration

Introduction

Magnetic nanoparticles with diameters less than 100 nm have attracted increasing interest as particles in this size range may allow investigation of fundamental aspects of magnetic ordering phenomena in magnetic materials with reduced dimensions and could lead to new technological applications [1-5]. Studies of magnetic nanoparticles have focused on the development of novel synthetic methods [5]. A nanocomposite is a material composed of two or more phases, one of which has a grain size of less than 100 nm. The combination of different physical or chemical properties may give rise to completely new materials [6,7]. It has been demonstrated that the formation of a passive coating of an inert material on the surface of iron oxide nanoparticles can help to improve their chemical stability and prevent their aggregation in liquids [8-11]. Recently, composite nanoparticles based on magnetic iron oxide have been prepared [12-16]. Such

magnetic nanocomposites have applications ranging from ferrofluids to separation science and technology [17].

In previous work, we described a method to prepare magnetic nanoparticles using a chemically induced transition [15,16,18,19] and Ni-Fe bixide composite nanoparticles were prepared using this method. In the preparation, a precursor consisting of FeOOH wrapped in $\text{Ni}(\text{OH})_2$ was synthesized by the well-known co-precipitation method. Then, using heat treatment in 0.25 M FeCl_2 solution at 100°C , a transition took place in which in addition to the $\text{Ni}(\text{OH})_2$ partially dissolving, the $\text{FeOOH}/\text{Ni}(\text{OH})_2$ precursor was transformed into $\gamma\text{-Fe}_2\text{O}_3/\text{Ni}_2\text{O}_3$ composite nanoparticles coated with FeCl_3 [15]. The Ni_2O_3 is weakly ferromagnetic [16] and the FeCl_3 is paramagnetic. Experiments have shown that such Ni-Fe bixide composite nanoparticles are very suitable for the synthesis of ferrofluids [20]. This chemically induced transition using FeCl_2 solution may provide a new route for the preparation of oxide nanoparticles. In the present work, we have investigated the characteristics of Ni-Fe bixide composite nanoparticles as a function of the concentration of FeCl_2 treatment solution.

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Experimental

Preparation

The preparation of the Ni-Fe bioxide composite nanoparticles was divided into two steps. Firstly, the precursor based on FeOOH wrapped with Ni(OH)₂ was synthesized using the co-precipitation method, which has been described in detail elsewhere [15,21]. The second step was to add the precursor to FeCl₂ solution, using concentrations of 0.025 M, 0.050 M, 0.075 M, 0.100 M, 0.125 M, 0.250 M, 0.500 M, 0.750 M and 1.000 M, to obtain 400ml of the mixed solution. Then this solution was heated to boiling point for 30 min in atmosphere; the nanoparticles precipitated gradually after the heating had stopped. Finally, these particles were dehydrated with acetone and allowed to dry naturally.

Characterization

A series of Ni-Fe oxide composite nanoparticles was prepared by a chemically induced transition involving FeCl₂ solution. The dependence on the concentration of the FeCl₂ solution was investigated by measuring the specific magnetization curves of the samples at room

temperature using a vibrating sample magnetometer (VSM, HH-15, applied field up to 10⁴ Oe).

The samples were prepared using FeCl₂ solutions 0.025 M, 0.075 M, 0.100 M, 0.125 M and 0.500 M, which were named samples (1), (2), (3), (4) and (5), respectively. In addition to the magnetic measurements, their crystal structures, morphology and chemical composition were analyzed by X-ray diffraction (XRD, XD-2, Cu K α radiation), transmission electron microscopy (TEM, JEM-2100E, at 100 kV) and X-ray photoelectron spectroscopy (XPS, Thermo ESCA250, Mg target).

Results and analysis

Figure 1 shows the specific magnetization curves of the samples. Clearly, all samples exhibited ferromagnetic features, with their specific magnetization varying non-monotonically with the concentration of FeCl₂ solution. At first, the magnetization strengthened as the FeCl₂ concentration increased from 0.025 M to 0.100 M, then the magnetization weakened as the concentration increased from 0.100 M to 1.000 M.

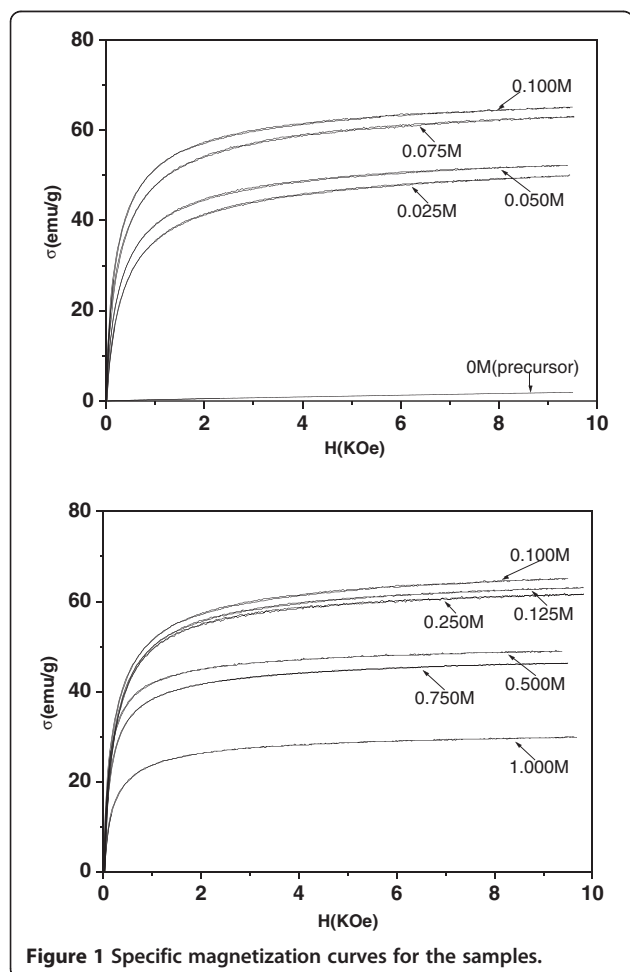


Figure 1 Specific magnetization curves for the samples.

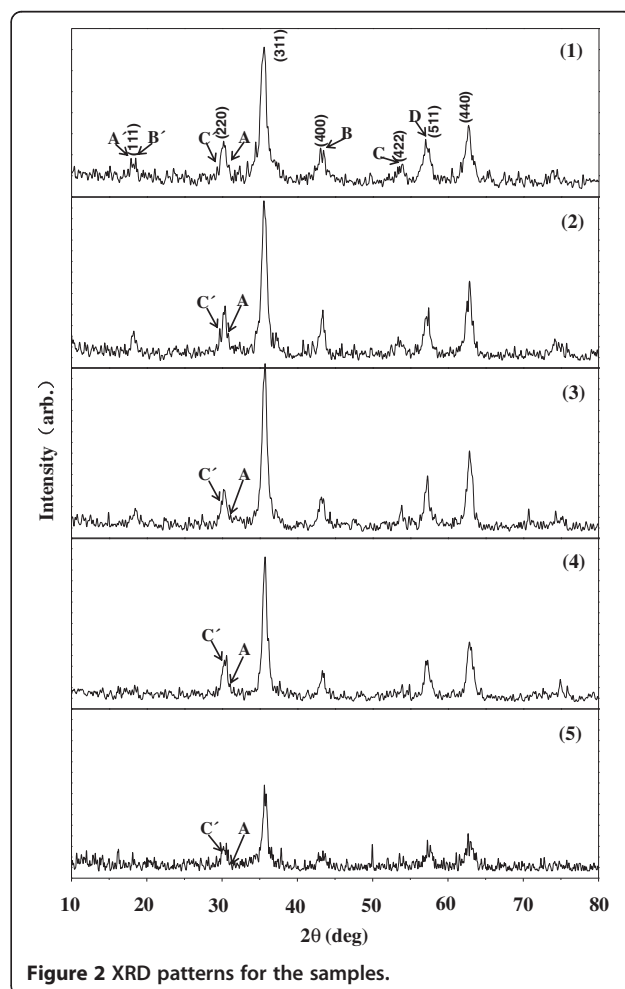


Figure 2 XRD patterns for the samples.

The XRD patterns of the samples are shown in Figure 2. The results show that these samples contain mainly γ -Fe₂O₃ with a trace of Ni₂O₃ and 2FeCl₃·5H₂O, as indicated by the arrows A, B, C and D for Ni₂O₃, and by arrows A', B' and C' for 2FeCl₃·5H₂O. For the ferrite nanoparticles, the grain sizes d_c can be estimated from the half-maximum width of the (311) diffraction peak β using Scherr's formula [22], $d_c = K\lambda/\beta\cos\theta$, where K is a constant 0.89, λ is the X-ray wavelength (Cu $K_\alpha = 0.1542$ nm) and θ is the Bragg diffraction angle of the (311) plane. The calculated results gave about the same value 8 nm for the γ -Fe₂O₃ grains in all the samples. In addition, comparing the intensity ratios of the A peak of Ni₂O₃ ($d = 0.2800$ nm) with the C' peak of 2FeCl₃·5H₂O ($d = 0.2980$ nm) show that the proportion

of Ni₂O₃ was reduced and 2FeCl₃·5H₂O increased as the concentration of the FeCl₂ solution increased.

TEM observations of the samples are shown in Figure 3. These results show that the particles in samples (1), (2), (3) and (4) are nearly spherical, with an average particles size d_p of about 11 nm, but in sample(5) there are a few rod-shaped particles (shown in the insert) in addition to the spherical particles. Clearly, the size of the spherical particles in sample(5) is less than those of samples (1), (2), (3) and (4), and is about 8 nm. High-resolution TEM results reveal that the particles have core-shell structure, as Figure 4 shown.

XPS measurements confirmed that there were Fe, O, Ni and Cl in the samples as illustrated in Figure 5. By

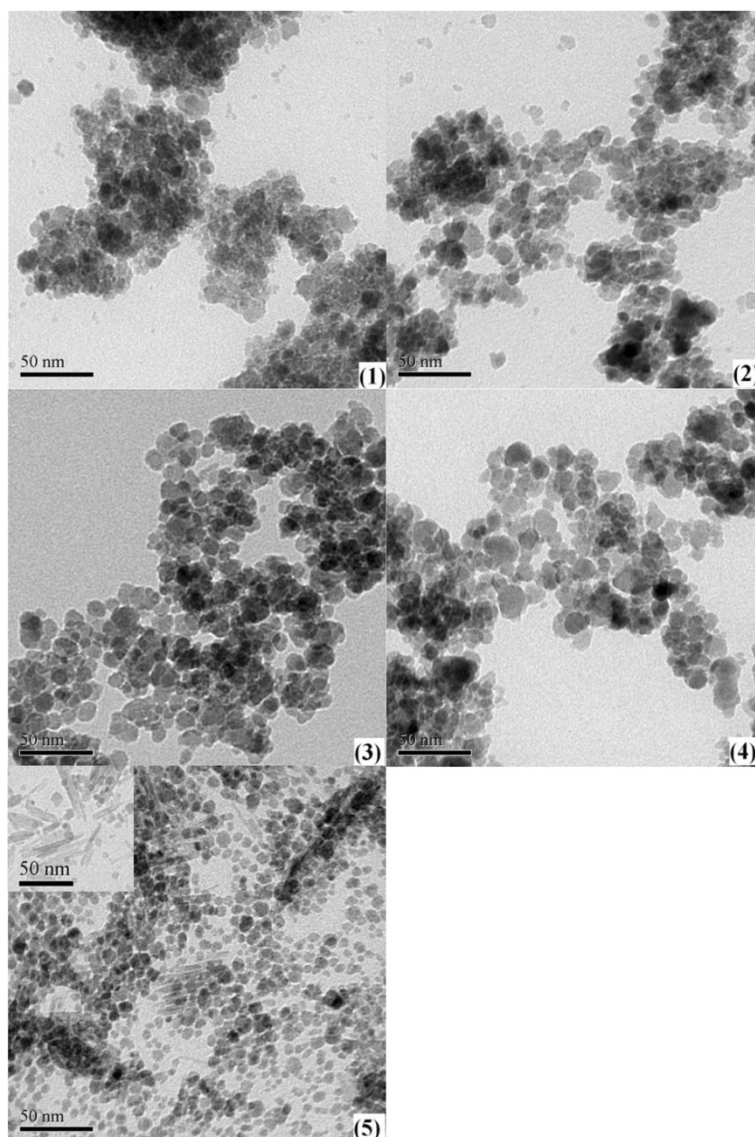
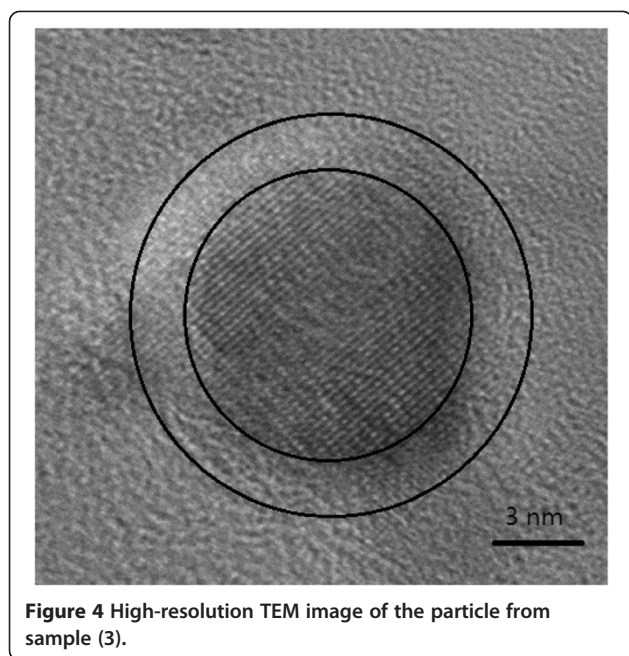


Figure 3 TEM images for the samples.



analysis of the binding energies in the spectra, it can be deduced that the samples consisted of Fe_2O_3 , Ni_2O_3 and FeCl_3 . The binding energy data are listed in Table 1. A quantitative analysis shows that for samples (1), (2), (3) and (4), the ratio Ni:Cl decreased in that order, the ratio Fe:Ni clearly increased and the ratio Fe:Cl increased slightly. For sample (5), the ratio Fe:Cl was clearly lower than that for sample (4), along with the ratio Ni:Cl, The ratio Fe:Ni was, however, higher. The complete data are listed in Table 2. In conclusion, it can be determined that for all the samples, the proportion of Ni_2O_3 phase decreased and FeCl_3 phase increased as the concentration of FeCl_2 solution increased. This also agrees with the XRD results.

Discussion

The results show that in the preparation of Ni-Fe bioxide nanoparticles, when the concentration of FeCl_2 solutions were less than 0.5 M, the samples (1), (2), (3) and (4) were single spherical particles consisting of a $\gamma\text{-Fe}_2\text{O}_3$ core, Ni_2O_3 outside the core and with an outermost layer of $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$. However, when the concentration reached 0.5 M, sample (5) formed rod-shaped particles together with spherical particles than smaller those of samples (1), (2), (3) and (4).

For samples (1), (2), (3) and (4), the results of both XRD analysis and TEM observations indicated that the $\gamma\text{-Fe}_2\text{O}_3$ grain size and the size of the complete particles remain about constant. However, the XPS results showed that the proportion of Ni_2O_3 decreased and FeCl_3 increased smoothly. This suggests that over the XPS detection range d_x determined by the mean free

paths of the electrons detected [23,24] and which is about 3 nm, the volume fraction of the $\gamma\text{-Fe}_2\text{O}_3$ phase remained constant (i.e. diameter of $\gamma\text{-Fe}_2\text{O}_3$ core d_r can be regarded as constant), as did the sum of the volume fractions of Ni_2O_3 and $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$, whose detection ranges are d_{Ni} and d_{Cl} , respectively. So, from samples (1) to (4), the reduction of Ni species and the increase of Cl species in the XPS results corresponds to the variation of the volume fraction, a thinning of the Ni_2O_3 layer and a thickening of the $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$ layer. This is also in agreement with the clear increase of the Fe:Ni ratio and the slight increase of the Fe:Cl ratio as the concentration of FeCl_2 solution increases. Since samples (1), (2), (3) and (4) consist of spherical particles and the measured XPS depth d_x is greater than the combined thickness of Ni_2O_3 (d_{Ni}) and FeCl_3 (d_{Cl}), as shown in Figure 6, the measured atomic ratio between Ni and Cl species Ni/Cl allows the molar ratio between Ni_2O_3 and FeCl_3 to be deduced as $\text{Ni}_2\text{O}_3/\text{FeCl}_3 = 1.5 \text{ Ni/Cl}$. The results are also listed in Table 2.

For sample (5), the results from both the XRD analysis and TEM observations show that the $\gamma\text{-Fe}_2\text{O}_3$ grain size is the same and the particles size is less than the samples (1), (2), (3) and (4), and the both sizes are about the same. Since there is much less Ni species than Fe, it is concluded that the spherical particles could consist of a $\gamma\text{-Fe}_2\text{O}_3$ core and a Ni_2O_3 surface layer. The average particle size depends mainly on the $\gamma\text{-Fe}_2\text{O}_3$ phase, and the rod-shaped particles may consist of crystals of $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$. This is also in agreement with the Fe:Cl ratio for samples (1) to (4) which shows a decrease rather than an increase.

In summary, as the concentration of FeCl_2 solution used for the chemically induced transition increases, the samples retain a constant $\gamma\text{-Fe}_2\text{O}_3$ composition but the proportion of Ni_2O_3 is reduced and that of $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$ increases. Clearly, the non-monotonic variation of the specific magnetization of the samples as a function of FeCl_2 concentration can be attributed to the phase changes. These can be formulated as follows.

The specific magnetization of the samples σ can be described as

$$\sigma = \phi_{m,\gamma}\sigma_\gamma + \phi_{m,\text{Ni}}\sigma_{\text{Ni}} + \phi_{m,\text{Cl}}\sigma_{\text{Cl}} \quad (1)$$

where σ_γ , σ_{Ni} , and σ_{Cl} are specific magnetizations, and $\phi_{m,\gamma}$, $\phi_{m,\text{Ni}}$ and $\phi_{m,\text{Cl}}$ are the mass fractions of the $\gamma\text{-Fe}_2\text{O}_3$, Ni_2O_3 and $2\text{FeCl}_3\cdot 5\text{H}_2\text{O}$ phases, respectively. According to the definition of the mass fraction, the relationship between $\phi_{m,\gamma}$, $\phi_{m,\text{Ni}}$ and $\phi_{m,\text{Cl}}$ is $\phi_{m,\gamma} + \phi_{m,\text{Ni}} + \phi_{m,\text{Cl}} = 1$. So, formula (1) can be written as

$$\sigma = \sigma_\gamma - \left[\phi_{m,\text{Ni}}(\sigma_\gamma - \sigma_{\text{Ni}}) + \phi_{m,\text{Cl}}(\sigma_\gamma - \sigma_{\text{Cl}}) \right] \quad (2)$$

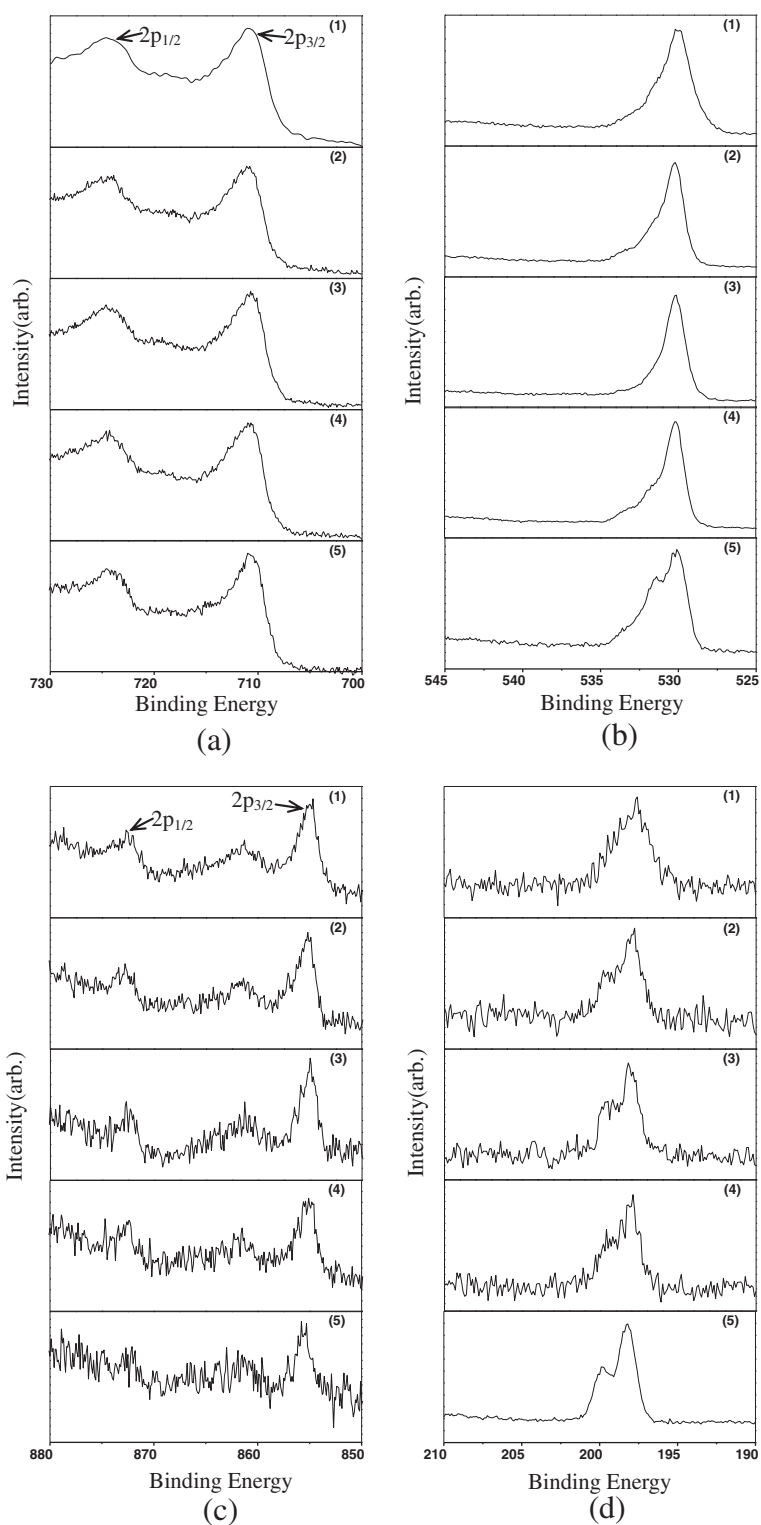


Figure 5 XPS results: Fe2p(a), O1s(b), Ni2p(c) and Cl1s(d).

Table 1 Binding energy data for the elements of the samples from XPS(eV)

	Fe2p _{3/2}	O1s	Ni2p _{3/2}	Cl2p
(1)	710.50	530.04	854.93	197.66
(2)	710.66	530.13	855.10	197.73
(3)	710.36	530.11	854.96	198.08
(4)	710.43	530.04	855.04	197.95
(5)	710.86	530.34	855.66	198.35
Fe ₂ O ₃	710.70	529.80		
Ni ₂ O ₃		531.80	855.60	
FeCl ₃	711.08			198.72

Note. The standard data for Fe₂O₃, Ni₂O₃, and FeCl₃ are taken from the HANDBOOK OF X-RAY PHOTOELECTRON SPECTROSCOPY By C. D. Wanger, W. M. Riggs, L. E. Davis, J. F. Moulder, G. E. Muilenberg (Editor).

In addition, the $\phi_{m, Ni}$ and $\phi_{m, Cl}$ can be described as follows

$$\phi_{m, Ni} = \frac{\rho_{Ni}}{\rho_{Ni} - \rho_{Cl} + [\phi_{v, \gamma}(\rho_{\gamma} - \rho_{Cl}) + \rho_{Cl}]} / \phi_{v, Ni}$$

$$\phi_{m, Cl} = \frac{\rho_{Cl}}{\rho_{Cl} - \rho_{Ni} + [\phi_{v, \gamma}(\rho_{\gamma} - \rho_{Ni}) + \rho_{Ni}]} / \phi_{v, Cl}$$

(3)

where ρ_{γ} , ρ_{Ni} and ρ_{Cl} are the densities, and $\phi_{v, \gamma}$, $\phi_{v, Ni}$ and $\phi_{v, Cl}$ are the volume fractions of γ -Fe₂O₃, Ni₂O₃ and 2FeCl₃·5H₂O, respectively, and $\phi_{v, \gamma} + \phi_{v, Ni} + \phi_{v, Cl} = 1$. From the experimental results, it is clear that $\phi_{v, \gamma}$ can be regarded as constant for all the samples. Thus, it can be determined from equation (3) that the variations of $\phi_{m, Ni}$ and $\phi_{m, Cl}$ depend on $\phi_{v, Ni}$ and $\phi_{v, Cl}$, respectively. In addition, the γ -Fe₂O₃ is ferrimagnetic, Ni₂O₃ is weakly magnetic and 2FeCl₃·5H₂O is paramagnetic, so that the magnetization of the samples depends mainly on the γ -Fe₂O₃ phase. Therefore, since $\phi_{m, Ni}(\sigma_{\gamma} - \sigma_{Ni}) \gg \phi_{m, Cl}(\sigma_{\gamma} - \sigma_{Cl})$, equation (2) can be written as

$$\sigma \cong \sigma_{\gamma} - [\phi_{m, Ni}(\sigma_{\gamma} - \sigma_{Ni})]$$

(4)

So, for concentrations of FeCl₂ solution below 0.100 M, as the concentration increases from 0.025 to 0.100 M, the $\phi_{m, Ni}$ (or $\phi_{v, Ni}$) decreases gradually, so

Table 2 The atomic percentages of Fe, O, Ni and Cl from XPS measurement and the molar ratio of Ni2O3/FeCl3

	Fe	O	Ni	Cl	Fe : Ni : Cl		Ni ₂ O ₃ / FeCl ₃
(1)	13.79	72.50	8.56	5.15	1	0.62	0.37
(2)	15.48	74.94	5.88	4.58	1	0.38	0.30
(3)	19.21	71.06	4.15	5.58	1	0.22	0.29
(4)	16.74	75.45	3.11	4.70	1	0.19	0.28
(5)	18.71	67.21	1.84	12.25	1	0.10	0.65

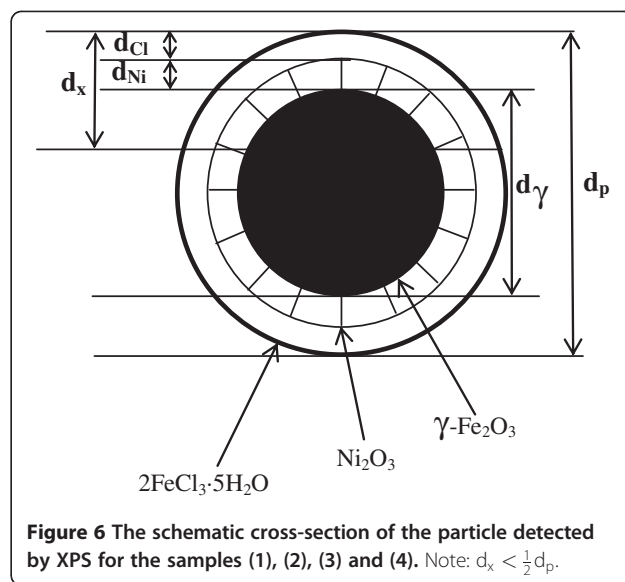


Figure 6 The schematic cross-section of the particle detected by XPS for the samples (1), (2), (3) and (4). Note: $d_x < \frac{1}{2}d_p$.

that σ increases. As long as $\phi_{m, Ni}(\sigma_{\gamma} - \sigma_{Ni}) \ll \phi_{m, Cl}(\sigma_{\gamma} - \sigma_{Cl})$, formula (2) can be written as

$$\sigma \cong \sigma_{\gamma} - [\phi_{m, Cl}(\sigma_{\gamma} - \sigma_{Cl})]$$

(5)

Therefore, for FeCl₂ solutions above 0.100 M, as the concentration increases from 0.100 to 1.000 M, the values of $\phi_{m, Cl}$ (or $\phi_{v, Cl}$) increase so that σ is reduced. In addition, it can be deduced that when the concentration of FeCl₂ solution is about 0.100 M, corresponding to sample(3), perhaps $\phi_{m, Ni}(\sigma_{\gamma} - \sigma_{Ni}) \cong \phi_{m, Cl}(\sigma_{\gamma} - \sigma_{Cl})$, i.e. $\phi_{m, Ni} / \phi_{m, Cl} \cong (\sigma_{\gamma} - \sigma_{Cl}) / (\sigma_{\gamma} - \sigma_{Ni})$, so the specific magnetization σ has its maximum value.

Conclusion

Using a chemically induced transition, Ni₂O₃- γ -Fe₂O₃ bixide composite nanoparticles can be prepared using FeCl₂ solutions with different concentrations. Using a number of characterization tools, such as VSM, XRD, TEM and XPS, the dependence of the samples on the concentration of the FeCl₂ solution has been revealed. When the FeCl₂ concentration was less than 0.500 M, the samples consisted of spherical Ni₂O₃- γ -Fe₂O₃ particles, about 11 nm diameter, coated with 2FeCl₃·5H₂O. When the FeCl₂ concentration was 0.500 M, the product consisted of both Ni₂O₃- γ -Fe₂O₃ spherical particles, of about 8 nm size, and 2FeCl₃·5H₂O rod-shaped particles. Nevertheless, the size of the γ -Fe₂O₃ grains was about the same for all samples. Significantly, the magnetization of the samples exhibited a non-monotonic variation although the ratio between the Ni and Cl species decreased monotonically with the increasing concentration of the FeCl₂ solution. It was noticed that samples prepared using FeCl₂ solutions with concentrations

0.025 M, 0.075 M, 0.100 M and 0.125 M, have the same size particles, about 11 nm, and same size of γ -Fe₂O₃ grains, about 8 nm. Therefore, it is deduced that the variation of the apparent magnetization has resulted from the competition between the reduced Ni₂O₃ phase and increasing 2FeCl₃·5H₂O. When the concentration of FeCl₂ solution does not exceed 0.100 M, the magnetization of the samples increases with increasing concentration since the rate of reduction of Ni₂O₃ is larger than the increase of 2FeCl₃·5H₂O. When the FeCl₂ concentration exceeds 0.100 M, the magnetization of the samples weakens since the increase of 2FeCl₃·5H₂O is now larger than the decrease of Ni₂O₃. Therefore, it can be concluded that using the chemically induced transition method to prepare Ni-Fe bixide composite nanoparticles, as long as the concentration of the FeCl₂ solution does not exceed 0.100 M, the thickness of both Ni₂O₃ and 2FeCl₃·5H₂O layers can be controlled and the γ -Fe₂O₃ core size remains constant. As a result, magnetic nanoparticles with a fixed size of about 11nm but different magnetization can be obtained.

Competing interests

Non-financial competing interests.

Authors' contributions

All authors contributed equally to this work. All authors read and approved the final manuscript.

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