Preparation of Magnetic Nanoparticles via a Chemically Induced Transition: Presence/Absence of Magnetic Transition on the Treatment Solution Used

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1. Introduction

Nanotechnology involves the understanding and control of matter with dimensions of roughly 1–100 nm [1]. Magnetic nanoparticles have attracted increasing interest because their size range enables the investigation of the fundamental aspects of magnetic-ordering phenomena in materials with reduced dimensions, and this has the potential to lead to new technological applications [2, 3]. Studies on magnetic nanoparticles have focused on the development of simple and effective methods for the fabrication of nanoparticles with controlled size, morphology, and properties [4, 5]. Iron oxide nanoparticles including magnetite ($\text{Fe}_3\text{O}_4$), hematite ($\alpha\text{-Fe}_2\text{O}_3$), maghemite ($\gamma\text{-Fe}_2\text{O}_3$), goethite ($\alpha\text{-FeOOH}$), and akaganeite ($\beta\text{-FeOOH}$) nanoparticles have attracted enormous attention due to their interesting properties [6–10]. Many methods have been developed for the preparation of $\gamma\text{-Fe}_2\text{O}_3$ magnetic nanoparticles, including coprecipitation [11], a gas-phase reaction technique [12], direct thermal decomposition [13], thermal decomposition-oxidation [14], sonochemical synthesis [15], a microemulsion technique [16], hydrothermal synthesis [17], a vaporization-condensation method [18], and a sol-gel approach [19].

Reactions for the synthesis of oxide nanoparticles using the coprecipitation method can be grouped into two categories. In the first category, the oxide is produced directly; in the second category, a precursor is produced initially and then subjected to further processing (drying, calcination, and subsequent steps) [20]. During the chemical reaction, which is followed by calcination or annealing, a new phase is formed. In addition to the transition from amorphous to crystalline, the particle size increases, and the crystallites aggregate as the calcination temperature increases [21]. The conventional aqueous synthesis of $\gamma\text{-Fe}_2\text{O}_3$ particles involves three or more steps [22], but we recently found a new route for the production of $\gamma\text{-Fe}_2\text{O}_3$ magnetic nanoparticles that requires
only two steps. This method involves the preparation of the paramagnetic FeOOH/Mg(OH)$_2$ precursor via a coprecipitation method, followed by treatment of the hydroxide precursor in the liquid phase using a ferrous chloride (FeCl$_2$) solution [23]. During this treatment, Mg(OH)$_2$ dissolves, and the paramagnetic FeOOH precursor transforms into ferrimagnetic γ-Fe$_2$O$_3$ nanoparticles via dehydration:

$$\text{Mg(OH)}_2 \rightarrow \text{Mg}^{2+} + 2\text{OH}^- \quad (1)$$
$$2\text{FeOOH (paramagnetic)} \rightarrow \gamma\cdot\text{Fe}_2\text{O}_3 \text{ (ferrimagnetic)} + \text{H}_2\text{O} \quad (2)$$

This method is referred to as a chemically induced transition [24, 25]. However, it remains unclear whether such a magnetic transition can be produced using other solutions. In the present work, we used aqueous solutions of ferric chloride (FeCl$_3$), cuprous chloride (CuCl), ferrous sulfate (FeSO$_4$), and ferrous chloride (FeCl$_2$) as treatment solutions to investigate the dependence of the magnetic transition on the treatment solution used and to attempt to explain the mechanism of the transition in the liquid phase.

2. Materials and Methods

2.1. Chemicals. FeCl$_3$, Mg(NO$_3$)$_2$, NaOH, CuCl, FeSO$_4$, and FeCl$_2$ of analytical grade and all other chemicals were used as received without further purification. Distilled water was used throughout the experiments.

2.2. Preparation. The precursor was synthesized via coprecipitation. An aqueous mixture of FeCl$_3$ (1 M, 40 mL) and Mg(NO$_3$)$_2$ (2 M, with 0.05 mol HCl; 10 mL) was added to an aqueous NaOH solution (0.7 M, 500 mL). The resulting solution was then heated to boiling for 5 min under stirring. The red-brown precursor precipitated gradually during this process. The precursor was collected and washed with dilute HNO$_3$ solution (0.01 M) until the pH of the supernatant liquid was 7-8.

The as-prepared precursor was mixed with the treatment solution (0.25 M, 400 mL), and the resulting mixture was allowed to boil for 30 min. The products were then dehydrated with acetonite and allowed to air-dry. Treatment with FeCl$_3$, CuCl, and FeSO$_4$ solutions produced samples (1), (2), and (3), respectively. For comparison, treatment was also performed in an FeCl$_2$ solution to produce sample (0).

2.3. Characterization. Crystal structures and specific magnetization curves were obtained for samples (0)–(3) using X-ray diffraction (XRD; XRD-7000, Shimadzu, Japan) and vibrating-sample magnetometry (VSM; HH-15, Nju-yq, China) at room temperature, respectively. The bulk chemical species, surface chemical composition, and morphology of samples (0) and (3) were determined using energy-dispersive X-ray spectroscopy (EDS; Quanta-200, Genesis, USA), X-ray photoelectron spectroscopy (XPS; XSAM800, Krator, UK), and transmission electron microscopy (TEM; Tecnai G20 ST, FEI, USA), respectively.

### Table 1: Atomic percentages ($\alpha_i$) determined from EDS measurements performed on samples (0) and (3).

<table>
<thead>
<tr>
<th></th>
<th>$\alpha_O$</th>
<th>$\alpha_{Fe}$</th>
<th>$\alpha_{Cl}$</th>
<th>$\alpha_S$</th>
<th>$\alpha_{Mg}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample (0)</td>
<td>60.48</td>
<td>37.93</td>
<td>1.59</td>
<td>3.93</td>
<td>1.37</td>
</tr>
<tr>
<td>Sample (3)</td>
<td>62.35</td>
<td>32.35</td>
<td>1.37</td>
<td>3.93</td>
<td>1.37</td>
</tr>
</tbody>
</table>

3. Results and Discussion

3.1. Results. XRD spectra are shown in Figure 1 for all of the samples. The crystal structure of samples (1) and (2) was different from that of sample (0), whereas the main structure of sample (3) is identical to that of sample (0), which corresponded to ferrite-like γ-Fe$_2$O$_3$. The results showed that sample (3) contained MgSO$_4$·5H$_2$O and Fe$_3$(SO$_4$)$_2$·11H$_2$O. The crystal phase of samples (1) and (2) is difficult to be distinguished from the XRD spectra, but it can be determined that both did not contain ferrite-like phase. Specific magnetization curves measured at room temperature are shown for all samples in Figure 2. The curves for samples (1) and (2) exhibited similar, apparently paramagnetic, behavior. Similar to sample (0), sample (3) appeared to be ferromagnetic having coercivity (see the windows in Figure 2), but its magnetization was weaker than that of sample (0). According to the XRD and VSM results, samples (1) and (2) showed no magnetic transition; in contrast, sample (3) exhibited a transition similar to that shown by sample (0). The specific saturation magnetization values ($\sigma_i$) for samples (0) and (3) were determined from a plot of $\sigma$ versus $1/H$ (where $H$ is the strength of magnetic field) in the high-field region, which is linear for strong magnetic fields [26]. $\sigma$ versus $1/H$ ($H \geq 7.5$ kOe) plots were represented as inserts of Figure 2, and the $\sigma_i$ values were determined by extrapolating plots to $\sigma$-axis to be 59.20 and 43.36 emu/g for samples (0) and (3), respectively.

Figure 3 shows EDS spectra for samples (0) and (3). Sample (0) contained Fe, O, and Cl, and sample (3) contained S and Mg, in addition to Fe and O. The quantitative results are listed in Table 1. XPS measurements revealed the same chemical species that were identified by the EDS analysis in samples (0) and (3). The XPS spectra are shown in Figure 4. The XRD and EDS results suggested that the γ-Fe$_2$O$_3$ phase was dominant in samples (0) and (3). There was also a Cl-containing phase in sample (0), and two S-containing phases in sample (3). It is possible that the Cl-containing phase in sample (0) was FeCl$_3$·6H$_2$O [25], but the content of FeCl$_3$·6H$_2$O may be so low that it did not produce clear diffraction peaks in the XRD spectrum (see Figure 1). The XRD results also implied that the S-containing phases in sample (3) were MgSO$_4$·5H$_2$O and Fe$_3$(SO$_4$)$_2$·11H$_2$O. Accordingly, the Fe$_{2p_{3/2}}$ lines and O$_{1s}$ lines observed for the two samples and the S$_{2p_{3/2}}$ line observed for sample (3) may be associated with two or more species. Detailed data on binding energies are listed in Table 2. We deduced from the experimental results that the binding energy of MgI in MgSO$_4$·5H$_2$O was approximately 1304.9 eV. In addition, it is noticed that the ferrite-like spinel structure, γ-Fe$_2$O$_3$ and Fe$_3$O$_4$, is difficult to discriminate by XRD.
Figure 1: XRD patterns of all samples, with (hkl), (hkl)$_{Cl}$, (hkl)$_{Mg}$, and (hkl)$_{Fe}$ corresponding to $\gamma$-Fe$_2$O$_3$, FeCl$_3$·6H$_2$O, MgSO$_4$·5H$_2$O, and Fe$_2$(SO$_4$)$_3$·11H$_2$O, respectively.

due to peak broadening [27] and by XPS because the data are very close (see Table 2). However, Fe$_3$O$_4$ is not very stable and is sensitive to oxidation [28]. It was found that Fe$_3$O$_4$ nanocrystallites transformed into $\gamma$-Fe$_2$O$_3$ nanocrystallites using ferric nitrate treatment [29]. Therefore, it is judged that the magnetic phase for samples (0) and (3) is $\gamma$-Fe$_2$O$_3$, rather than Fe$_3$O$_4$ or mixed phase of both $\gamma$-Fe$_2$O$_3$ and Fe$_3$O$_4$.

TEM observations revealed that sample (3) was identical to sample (0) and that it consisted of nearly spherical nanoparticles. Typical TEM images for both samples are shown in Figure 5. Statistical analysis [30] indicated that the diameter of the nanoparticles fitted a lognormal distribution. The median diameters ($d_g$) for samples (0) and (3) were 10.55 and 13.16 nm, respectively, and the associated standard
Figure 2: Specific magnetization curves measured at room temperature for all samples.

Figure 3: EDS spectra for samples (0) and (3).
Figure 4: XPS spectra for samples (0) and (3).
deviation values (ln $\sigma_g$, where $\sigma_g$ is the geometry deviation) were 0.37 and 0.31, respectively.

3.2. Discussion. Experimental XRD and magnetization curve measurements indicated that the products after treatment with an FeCl$_3$ solution were not ferromagnetic. The product after treatment with an FeSO$_4$ solution exhibited a magnetic transition, similar to that observed for the products generated after treatment with an FeCl$_2$ solution. These products mainly consisted of a ferrimagnetic $\gamma$-Fe$_2$O$_3$ phase. These results show that the magnetic transition was induced by Fe$^{2+}$, rather than by the acid radical ions. Additionally, samples (0) and (3) contained FeCl$_3$·6H$_2$O and Fe$_3$(SO$_4$)$_2$·11H$_2$O, which, respectively, correspond to the ferrous salts FeCl$_3$ and FeSO$_4$ treatment solution. This observation suggests that the ferrous salts induced the transformation of FeOOH in the precursor into $\gamma$-Fe$_2$O$_3$ via dehydration, and this resulted in the simultaneous oxidation of Fe$^{2+}$ to Fe$^{3+}$. The results for sample (1) show that the ferric salt did not induce a transition, suggesting that the oxidation of the ferrous ions in the liquid phase was necessary for the dehydration of amorphous FeOOH to form magnetic phase. The experimental results for sample (2) show that the cuprous ions (Cu$^{+}$), which may not have had the tendency to oxidize to cupric ions (Cu$^{2+}$) in the liquid phase [31], did not cause FeOOH to form magnetic phase. This implies that the dehydrating action of the cuprous ions was weaker than that of the ferrous ions in the chemically induced transition in the liquid phase.

The experimental results also indicated that the products after treatment with an FeSO$_4$ solution contained MgSO$_4$, but the products after treatment with an FeCl$_2$ solution had no corresponding Mg-containing constituent. This result suggests that FeSO$_4$ in the treatment solution not only induced the transformation of FeOOH into $\gamma$-Fe$_2$O$_3$ via dehydration and then underwent oxidation into Fe$_2$(SO$_4$)$_3$, but also reacted with dissolved Mg(OH)$_2$, producing MgSO$_4$:

$$\text{Mg}^{2+} + 4\left(\text{Fe}^{3+} + \text{SO}_4^{2-}\right) \rightarrow \text{MgSO}_4 + \text{Fe}_2\left(\text{SO}_4\right)_3 + 2\text{Fe}^{3+}$$

(3)

According to (3), MgSO$_4$ first adsorbed onto the $\gamma$-Fe$_2$O$_3$ crystallites, forming MgSO$_4$·5H$_2$O. Some Fe$^{3+}$ and SO$_4^{2-}$ ions also adsorbed, forming Fe$_3$(SO$_4$)$_2$·11H$_2$O on the outermost layer. Thus, the solutions of ferrous salts with different acid radicals (e.g., FeCl$_3$ and FeSO$_4$) could induce the transformation of the FeOOH/Mg(OH)$_2$ precursor into a $\gamma$-Fe$_2$O$_3$

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**Table 2: Binding energies (eV) of elements, determined from XPS measurements performed on samples (0) and (3).**

<table>
<thead>
<tr>
<th></th>
<th>Fe2p$_3/2$</th>
<th>O1s</th>
<th>Cl2p$_3/2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample (0)</td>
<td>710.81</td>
<td>530.19</td>
<td>198.84</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>710.9</td>
<td>530.2</td>
<td></td>
</tr>
<tr>
<td>Fe$_3$O$_4$</td>
<td>710.8</td>
<td>530.1</td>
<td></td>
</tr>
<tr>
<td>FeCl$_3$</td>
<td>711.3</td>
<td>199</td>
<td></td>
</tr>
<tr>
<td>Sample (3)</td>
<td>711.41</td>
<td>530.40 (P1); 531.65 (P2)</td>
<td>168.99</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>710.9</td>
<td>530.2</td>
<td></td>
</tr>
<tr>
<td>Fe$_3$O$_4$</td>
<td>710.8</td>
<td>530.1</td>
<td></td>
</tr>
<tr>
<td>Fe$_3$(SO$_4$)$_3$</td>
<td>711.05</td>
<td>531.6</td>
<td>169.1</td>
</tr>
</tbody>
</table>
| MgSO$_4$ | ? | 168.6 | ?

Note: the standard data are from the NIST online database for XPS at http://www.nist.gov; there are no O1s and Mg1s binding energies for MgSO$_4$. 

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Figure 5: Typical TEM images for samples (0) and (3).
**Table 3:** Molar and mass percentages of phases in samples (0) and (3).

<table>
<thead>
<tr>
<th>Molar percentage (y)</th>
<th>Mass percentage (z)</th>
</tr>
</thead>
<tbody>
<tr>
<td>y-Fe₂O₃/FeCl₃</td>
<td>y-Fe₂O₃/MgSO₄/Fe₂(SO₄)₃</td>
</tr>
<tr>
<td>Sample (0)</td>
<td>97.24/2.76</td>
</tr>
<tr>
<td>Sample (3)</td>
<td>87.34/7.81/4.85</td>
</tr>
</tbody>
</table>

The mass percentage values are listed in Table 3. The mass percentages of each phase (y) and (z) were calculated using the following expressions:

$$y_\gamma = \frac{a_{Fe} - 2(a_s - a_{Mg})/3}{a_{Fe}/2 + a_{Mg}} \times 100,$$

$$y_{Mg} = \frac{a_{Mg}}{a_{Fe}/2 + a_{Mg}} \times 100,$$

$$y_S = 100 - y_\gamma - y_{Mg},$$

where $$a_{Fe}$$, $$a_{Mg}$$, and $$a_s$$ are the atomic percentages of Fe, Mg, and S in sample (3), respectively. Thus, the molar percentages of each phase (y) in samples (0) and (3) could be calculated from the $$a_i$$ values, which were measured using EDS, and are listed in Table 3. The mass percentages of each phase (z) in a sample were deduced using the following expression:

$$z_i = \frac{y_i A_i}{\sum y_j A_j} \times 100,$$

where $$A_i$$ is the molar weight of the i-th phase. Accordingly, the mass percentage of each phase was calculated for samples (0) and (3) from the molar percentages (y) and molar weights of y-Fe₂O₃, FeCl₃⋅6H₂O, MgSO₄⋅5H₂O, and Fe₂(SO₄)₃⋅11H₂O. The mass percentage values are listed in Table 3. Table 3 indicates that the main species in samples (0) and (3) was y-Fe₂O₃. The $$\sigma_i$$ values for samples (0) and (3) could therefore be expressed as

$$\sigma_i \approx z_\gamma \frac{\sigma_{\gamma\sigma}}{100},$$

where $$z_\gamma$$ is the mass percentage of the y-Fe₂O₃ phase and $$\sigma_{\gamma\sigma}$$ is the specific saturation magnetization of the y-Fe₂O₃ phase. For samples (3) and (0), their $$\sigma_{\gamma\sigma}$$ can be regarded as same since the y-Fe₂O₃ phase resulted from the same FeOOH phase. Thus, the ratio of the $$\sigma_i$$ values for samples (3) and (0) was proportional to the mass fraction $$z_\gamma$$ of y-Fe₂O₃. From the experimental results for the magnetization, we found that the ratio of the $$\sigma_i$$ values for samples (3) and (0) was 0.73, which agreed with the ratio of the mass percentages $$z_i$$ of the y-Fe₂O₃ phase (0.79). As a consequence, the mass percentage was available.

The volume percentage of each phase of the samples ($$\Phi_i$$) could be obtained from the $$z_i$$ value:

$$\Phi_i = \frac{z_i/\rho_i}{\sum z_i/\rho_i} \times 100,$$

where $$\rho_i$$ is the density of the i phase. The $$\rho_i$$ values for y-Fe₂O₃, FeCl₃⋅6H₂O, MgSO₄⋅5H₂O, and Fe₂(SO₄)₃⋅11H₂O magnetic phase via dehydration. However, the coating on the y-Fe₂O₃ crystallites varied when the treatment solution was changed. The results revealed that sample (0) contained y-Fe₂O₃ and FeCl₃⋅6H₂O, and sample (3) contained y-Fe₂O₃, MgSO₄⋅5H₂O, and Fe₂(SO₄)₃⋅11H₂O. A schematic diagram illustrating the formation of the nanoparticles using FeCl₂ and FeSO₄ solutions is shown in Figure 6. As the magnetic nanoparticles have surface heterolayers that are different from the magnetic core, the magnetic core spins close to surface can be pinned by the layer, and the pinning would cause an unusually large coercivity [32, 33]. So, such y-Fe₂O₃ based magnetic nanoparticles appeared to be apparently ferromagnetic due to surface pinning rather than shape effects [34].

Because the metal salts were paramagnetic, the difference in the apparent magnetization of samples (0) and (3) depended on the y-Fe₂O₃ content. For sample (0), the molar percentages of y-Fe₂O₃ and FeCl₃⋅6H₂O ($$y_\gamma$$ and $$y_{Cl}$$, resp.) could be described as follows:

$$y_\gamma = \frac{(a_{Fe} - a_{Cl}/3)/2}{(a_{Fe} - a_{Cl}/3)/2 + a_{Cl}/3} \times 100,$$

$$y_{Cl} = 100 - y_\gamma,$$

where $$a_{Fe}$$ and $$a_{Cl}$$ are the atomic percentages of Fe and Cl in sample (0), respectively. For sample (3), the molar percentages of y-Fe₂O₃, MgSO₄⋅5H₂O, and Fe₂(SO₄)₃⋅11H₂O ($$y_\gamma$$, $$y_{Mg}$$, and $$y_S$$, resp.) could be described as follows:

$$y_\gamma = \frac{(a_{Fe} - 2(a_s - a_{Mg})/3)/2}{a_{Fe}/2 + a_{Mg}} \times 100,$$

$$y_{Mg} = \frac{a_{Mg}}{a_{Fe}/2 + a_{Mg}} \times 100,$$

$$y_S = 100 - y_\gamma - y_{Mg},$$

Figure 6: Schematic diagram of the formation of magnetic nanoparticles using FeCl₂ and FeSO₄ solutions.
Table 4: Volume percentages of phases ($\Phi_i$) in samples (0) and (3).

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\gamma$-Fe$_2$O$_3$/FeCl$_3$/6H$_2$O</th>
<th>$\gamma$-Fe$_2$O$_3$/MgSO$_4$/5H$_2$O/Fe$_2$(SO$_4$)$_3$/11H$_2$O</th>
</tr>
</thead>
<tbody>
<tr>
<td>(0)</td>
<td>88.69/11.31</td>
<td>56.18/17.07/26.75</td>
</tr>
<tr>
<td>(3)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

were 4.90, 1.844, 1.896, and 2.14 g/cm$^3$, respectively. The values calculated for $\Phi_i$ are listed in Table 4.

Because the particles in samples (0) and (3) were nearly spherical, the ratio of the average particle volume in sample (0) ($\langle v \rangle_{(0)}$) to the average particle volume in sample (3) ($\langle v \rangle_{(3)}$) could be described as follows:

$$
\frac{\langle v \rangle_{(0)}}{\langle v \rangle_{(3)}} = \frac{\langle d \rangle_{v,(0)}}{\langle d \rangle_{v,(3)}},
$$

where $\langle d \rangle_{v,(0)}$ and $\langle d \rangle_{v,(3)}$ are the diameters corresponding to the average volumes of the particles in samples (0) and (3), respectively. $\langle d \rangle_{v}$ could be calculated directly from the size distribution parameters obtained using TEM by using the equation $\langle d \rangle_{v} = \exp(\ln d_p + 1.5 \ln^2 \sigma_p)$ [30]. Accordingly, a $\langle d \rangle_{v,(0)}/\langle d \rangle_{v,(3)}$ value of 0.62 was obtained. Additionally, the average volume of the $\gamma$-Fe$_2$O$_3$ based nanoparticles, which contained many phases, ($\langle v \rangle$), can be expressed as $\langle v \rangle = \langle v \rangle_{(3)}$ $\Phi_{(3)}$ $\Phi_{(0)}$, where $\langle v \rangle_{(3)}$ is the average volume of the $\gamma$-Fe$_2$O$_3$ phase and $\Phi_{(3)}$ is the volume percentage of the $\gamma$-Fe$_2$O$_3$ phase. Because the $\gamma$-Fe$_2$O$_3$ phase was produced from FeOOH, the $\langle v \rangle_{(3)}$ values for samples (0) and (3) were the same. Therefore, the ratio of $\langle v \rangle_{(0)}$ to $\langle v \rangle_{(3)}$ could be described by $\Phi_{(3)}/\Phi_{(0)}$; that is,

$$
\frac{\langle v \rangle_{(0)}}{\langle v \rangle_{(3)}} = \frac{\Phi_{(3)}}{\Phi_{(0)}},
$$

where $\Phi_{(0)}$ and $\Phi_{(3)}$ are the volume percentages of the $\gamma$-Fe$_2$O$_3$ phase in samples (0) and (3), respectively. Thus, the $\langle v \rangle_{(0)}/\langle v \rangle_{(3)}$ value obtained from the volume percentages was 0.63, which agreed with the value of $\langle d \rangle_{v,(0)}/\langle d \rangle_{v,(3)}$ (0.62) and confirmed the validity of both the coating model of the nanoparticle structure and the volume percentage.

4. Conclusions

Metal ions in the treatment solution play a key role in the magnetic transition during the preparation of magnetic nanoparticles via the chemically induced transition of FeOOH/Mg(OH)$_2$. No magnetic transition occurred when FeCl$_3$ and CuCl were used as treatment solutions; in contrast, the product obtained using treatment with an FeSO$_4$ solution, which is similar to that obtained using an FeCl$_3$ solution, showed ferromagnetic behavior. The magnetic phase of the two products was $\gamma$-Fe$_2$O$_3$. Experimental results revealed FeCl$_3$ and Fe$_2$(SO$_4$)$_3$ in the products after treatment with FeCl$_3$ and FeSO$_4$ solutions, respectively. Thus, Fe$^{2+}$, which likely has a stronger dehydrating ability than the cuprous ion Cu$^{+}$, might have undergone oxidation to Fe$^{3+}$, thereby inducing a magnetic transition via dehydration. Treatment with an FeSO$_4$ solution resulted in the formation of MgSO$_4$ on the $\gamma$-Fe$_2$O$_3$ crystallites (the binding energy of Mg1s is $\sim$1304.9 eV), whereas no Mg-containing compound formed in the product of the treatment using an FeCl$_3$ solution. This shows that the nonmagnetic coating on the magnetic $\gamma$-Fe$_2$O$_3$ crystallites varied with changes in the acid radical ions in the treatment solution. Treatment with an FeSO$_4$ solution led to a higher content of the nonmagnetic phase in the product, and thus weaker magnetization, despite the fact that these particles were larger than those formed after treatment with an FeCl$_3$ solution. Due to the surface pinned effect, such $\gamma$-Fe$_2$O$_3$ based magnetic nanoparticles, which were produced via the chemically induced transition, exhibited apparently ferromagnetism.

Here, we demonstrated the use of an FeOOH/Mg(OH)$_2$ precursor and a ferrous salt treatment solution (as FeCl$_3$ solution or FeSO$_4$ solution) as a simple and effective method for the preparation of $\gamma$-Fe$_2$O$_3$-based magnetic nanoparticles. Obviously, the alkali-oxide FeOOH dehydrating into $\gamma$-Fe$_2$O$_3$ was induced with Fe$^{2+}$ in ferrous salt solution transforming into Fe$^{3+}$. For the preparation of magnetic nanoparticles via chemically induced transition (CIT) method, the relation between acting energy of the alkali-oxide dehydrating and oxidation of the ferrous ions is interesting, and this mechanism will be further investigated in future work.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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References


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