Synthesis of Fe3O4 nanoparticles from Ferric Nitrate by Sol-Gel Method

تخليق جزيئات النانوية لأكسيد الحديد المغناطيسي "Fe3O4" من نترات الحديدية بواسطة طريقة محلول - غروي

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Abstract:
Fe3O4 nanoparticles were synthesized from Ferric Nitrate by Sol-Gel method with annealing under vacuum. The phase structures, morphologies, particle sizes, chemical composition, and magnetic properties of Fe3O4 nanoparticles were characterized by TEM, U.V and XRD. The results indicated that the size, the corresponding saturation magnetization value and coercivity value of Fe3O4 nanoparticles increase with the increase of temperature. Fe3O4 nanoparticles was studied under different atmospheres and temperatures.

Keywords: Ferric Nitrate, Fe3O4 nanoparticles, properties and characterization.

الملخص:
خلقت جزيئات النانوية لأكسيد الحديد المغناطيسي من نترات الحديد بواسطة محلول -غروي مع درجة الحرارة العالية وتحت ضغط. الترakitيب الطوري، المركبونيات والأحجام الجزئية تراكيب الكيميائي ، الصفات المغناطيسية من جزيئات النانوية الأكسيد الحديد المغناطيسي "Fe3O4" دراسة التحليل .TEM، XRD وU.V بواسطة اجهزة اجهزة والنتائج تشير إلى الوراء التراث وقيمة المغناطيسية لواضع الارتباط والقیم.
Introduction:

In the past decade, a variety of methods have been developed to form highly structure-controlled materials of functionalized metals, semiconductors and copolymer nanoparticles on the nano- or microscale. As a versatile kind of material, magnetite has attracted much attention in recent years.

Ferrite iron (Fe₃O₄) is a traditional magnetic material used in magnetic storage media, solar energy transformation, electronics, ferrofluids, biomedicine and catalysis [1-4]. During the last decade, significant research has been done in the field of nanosized magnetic particles, due to their potential for biomedical applications, such as improving the quality of Magnetic Resonance Imaging (MRI), and drug delivery systems [5 – 6 ].

Fe₃O₄ nanoparticles have been the subject of intense interest because of their potential applications in several advance technological areas due to their promising physical and chemical properties. Generally, these properties depend on the size and structure of particles. Fe₃O₄ nanoparticles find wide applications in the field of biomedical, as anticancer agent and corrosion protective pigments in paints and coatings [7 - 9]. The magnetic atoms or ions in such solid materials are arranged in a periodic lattice and their magnetic moments collectively interact through molecular exchange fields, which give rise to a long-range magnetic ordering.

Among all iron oxide nanoparticles, Fe₃O₄ represent the most interesting properties due to of its unique structure i.e. the presence of iron cations in two valence states, Fe²⁺, Fe³⁺ on tetrahedral and octahedral sites with an inverse cubic spinel structure. The coercivity and remenance values for the super paramagnetic nano Fe₃O₄ nanoparticles have been found to be zero by the earlier reported methods [10-13]. Presently, cell labelling strategies find application of superparamagnetic ferrite either through conjugating the magnetic nanoparticles to the cellular surface of the stem cell or by internalization of the particles into the cell. Superparamagnetic ferrite can work in both of these ways, since the potential to manipulate their surface chemistry is plentiful and their sizes along
with other attributes promote their successful uptake into cells. The superparamagnetic nano ferrites also interface well with MRI technology.

The use of superparamagnetic particles play a crucial role in the diagnostic imaging modality technique finds application in the study of stem cell [14-15].

In this paper $\text{Fe}_3\text{O}_4$ nanoparticles were synthesized by heating to $40^\circ\text{C}$ at 2 hours and at $80^\circ\text{C}$ for 2 hours to obtained Sol Gel followed by drying for 6 hours at $120^\circ\text{C}$ and then annealing at $200^\circ\text{C}$ and $400^\circ\text{C}$ in oven under vacuum to obtain $\text{Fe}_3\text{O}_4$ powder.

**Experimental Materials :**

Ferric nitrate ($\text{Fe(NO}_3\text{)}_3.9\text{H}_2\text{O}$) and ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$) of analytical grade were obtained from Sinopharm Chemical Reagent Co ,Ltd ,China. The reagents were used without further purification.

Physical parameters of Ferric nitrate ($\text{Fe(NO}_3\text{)}_3.9\text{H}_2\text{O}$) and Glycol ($\text{C}_2\text{H}_6\text{O}_2$) are reported in table 1 and 2 respectively.

**Table 1. General Characteristics of Ferric nitrate ($\text{Fe(NO}_3\text{)}_3.9\text{H}_2\text{O}$)**

<table>
<thead>
<tr>
<th>Molecular formula</th>
<th>Ferric nitrate ($\text{Fe(NO}_3\text{)}_3.9\text{H}_2\text{O}$) $\geq$ 98.5 %</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Appearance</strong></td>
<td>White Powder</td>
</tr>
<tr>
<td><strong>Molecular weight</strong></td>
<td>404</td>
</tr>
<tr>
<td><strong>Company</strong></td>
<td>Sinopharm chemical reagent Co ,Ltd ,China</td>
</tr>
</tbody>
</table>

**Table 2. General Characteristics of Ethylene Glycol ($\text{C}_2\text{H}_6\text{O}_2$)**

<table>
<thead>
<tr>
<th>Molecular formula</th>
<th>Ethylene Glycol ($\text{C}_2\text{H}_6\text{O}_2$) $\geq$ 99 %</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Appearance</strong></td>
<td>liquid</td>
</tr>
<tr>
<td><strong>Molecular weight</strong></td>
<td>62.07</td>
</tr>
<tr>
<td><strong>density</strong></td>
<td>1.111- 1.115</td>
</tr>
<tr>
<td><strong>Company</strong></td>
<td>Sinopharm chemical reagent Co ,Ltd ,China</td>
</tr>
</tbody>
</table>
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Preparation of Magnetite Nanoparticles:

The procedure of synthesizing magnetite nanoparticles [16-19] is described as follows:

20.2 gram ferric nitrate was first dissolved in 25 mL ethylene glycol with vigorous stirring speed 500 r.p.m in 100 mL three necked flask glass with out cover for 2 hours at 40°C, and then the solution was heated to 80°C and kept at the temperature to obtain brown gel. The gel was obtained after 2 hours and then dried at 120°C for about 4 hours in petri dish. After drying, the xerogel was annealed at temperature range 200 and 400°C under vacuum. Finally, different size magnetite nanoparticles were obtained.

Transmission Electron Microscope (TEM) Test:

For TEM Test, a small amount of sample was dissolved in 3mL of deionized water in test tube and the solution was stirred by ultra-sonication. Then 10 µL sample was transferred to clean Copper Grid and kept for drying for TEM test. The TEM micrographs of samples were observed by CM 12 Philips Transmission Electron Microscope.

Results and Discussion:

Plate 1,2,3,4,5,6,7 and 8 (TEM) at at 200°C and plate 9,10,11,12,13,14,15,16,17,18 and 19 (TEM) at at 400°C shows the top-view TEM images of the Fe3O4 Nanoparticle plate (TEM). The size of the Fe3O4 nanoparticle is clear from the TEM. The surface of Fe3O4 nanoparticle shows several large meandering wrinkles. The size of Fe3O4 nanoparticle can be clear from TEM image. Fig (1 and 2) X-ray diffraction shown the graph all of Magnetite and Fe3O4 nanoparticle. Fig (3 and 4) U.V shown the graph all of Magnetite and Fe3O4 nanoparticle respectively dispersed in chloroform.
References:

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Plate 5: Fe₃O₄ 200 °C
Plate 6: Fe₃O₄ 200 °C
Plate 7: Fe₃O₄ 200 °C
Plate 8: Fe₃O₄ 200 °C
Plate 9: Fe₃O₄ 400 °C
Plate 10: Fe₃O₄ 400 °C
Plate 11: Fe₃O₄ 400 °C
Plate 12: Fe₃O₄ 400 °C
Plate 13: Fe₃O₄ 400 °C
Plate 14: Fe₃O₄ 400 °C
Plate 15: Fe₃O₄ 400 °C
Plate 16: Fe₃O₄ 400 °C
Plate 17: Fe₃O₄ 400 °C
Plate 18: Fe₃O₄ 400 °C
Fig. 1: XRD for Fe$_3$O$_4$ Nanoparticle at 200 $^\circ$C

Fig. 2: XRD for Fe$_3$O$_4$ Nanoparticle at 400 $^\circ$C
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Fig. 3: U.V of Fe3O4 200 °C

Fig. 4: U.V of Fe3O4 400 °C