Notice

DESCRIPTION CN103288140A

A simple method for controlling the morphology of iron oxide nanoparticles

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一种简单调控四氧化三铁纳米粒子形貌的方法

[0001]

Technical Field

技术领域

[0002]

This invention relates to a method for preparing iron(III) oxide, specifically a method for simply controlling the morphology of iron(III) oxide nanoparticles using a co-precipitation method.

本发明属于一种制备四氧化三铁的方法，具体来说涉及一种采用共沉淀法简 单调控四氧化三铁纳米粒子形貌的方法。

[0003]

Background Technology

背景技术

[0004]

As is well known, Fe3O4 (Fe3O4) nanoparticles have broad application prospects in magnetic recording materials, biomedicine, catalysis, adsorption separation and other fields due to

their advantages such as high saturation magnetization, low toxicity and side effects, good chemical stability and low cost, and have become a research focus of magnetic nanomaterials.

众所周知，四氧化三铁（Fe ₃ O ₄ ）纳米粒子由于具有高的饱和磁化强度、毒副 作用低、化学稳定性好、价廉等优点，在磁记录材料、生物医药、催化、吸附 分离等领域有广泛的应用前景，成为磁性纳米材料的研究重点。

The morphology of nanoparticles greatly influences their performance and application fields. Different morphologies of iron oxide nanoparticles exhibit different properties. Therefore, the study of the structure and morphology control of iron oxide nanoparticles has become one of the hot topics.

纳米粒子的形 貌在很大程度上影响着其性能与应用领域，不同形貌的四氧化三铁纳米粒子表 现出不同的性能，因此四氧化三铁纳米粒子的结构与形貌控制研究成为热点课 题之一。

[0005]

There are many methods for preparing iron oxide nanoparticles, commonly used methods include coprecipitation, hydrothermal method, solvent thermal method, and ultrasonic method. Nanomaterials in the shapes of nanoparticles, nanocubes, nanorods, and nanowires have been successfully prepared.

制备四氧化三铁纳米粒子的方法有很多种，常用的有共沉淀法、水热法、溶 剂热法、超声法等方法，已成功制备出了纳米颗粒、纳米立方体、纳米棒、纳 米线等形状的纳米材料。

Gao et al. prepared Fe₃O₄ nanocubes using a solvothermal method (Crystal Growth & Design, 10:2888-2894); Zhou et al. synthesized a series of Fe₃O₄ nanorods, nanooctahedrons, and nanocubes using a hydrothermal method (Journal of Solid State Chemistry, 196:138-144); Zhao Guizhe et al. disclosed a method for controlling the morphology of Fe3O4, using a hydrothermal method to achieve the regulation of the spherical, microporous, and hollow structures of Fe3O4 [Chinese Invention Patent, Application No.: 201210158461.2].

Gao等人采用溶剂热法制备了Fe ₃ O ₄ 纳米立方体（晶体 生长与设计，Crystal Growth&Design10:2888-2894）；Zhou等人采用水热法 合成了一系列Fe ₃

O ₄ 纳米棒、纳米八面体和纳米立方体（固体化学，

Journal of Solid State Chemistry196:138-144）；赵贵哲等人公开了一种控制四氧化三 铁形貌与形貌的方法，采用水热法实现了四氧化三铁球形、微孔和中空结构的 调控[中国发明专利，申请号：201210158461.2]。

These preparation methods involve complex processes, expensive raw materials, and high production costs.

这些制备方法的制备工艺较 为复杂，原料价格昂贵，生产成本高。

[0006]

Currently, there are no reports on the preparation of iron oxide nanocubes and nanorods using the co-precipitation method.

目前，未见有采用共沉淀法制备得到四氧化三铁纳米立方体和纳米棒的相关 报道。

[0007]

Summary of the Invention

发明内容

[0008]

To address the shortcomings of existing technologies, this invention provides a low-cost method for simply controlling the morphology of iron oxide nanoparticles using a co-precipitation method.

本发明为解决现有技术的不足之处，提供一种成本低的用共沉淀法简单调控 四氧化三铁纳米粒子形貌的方法。

[0009]

This invention is achieved through the following technical solution, characterized by including the following steps:

本发明是通过以下技术方案实现的，其特征在于包括以下步骤：

[0010]

(1) Dissolve ferrous salts and ferric salts in deionized water to prepare a solution with a concentration of 0.15-1.0 mol/L. Add solid NaOH to the solution to adjust the pH value to 10-

12. Stir at room temperature for 10-60 minutes to obtain a black precipitate of iron oxide. Filter the precipitate and wash it with deionized water and anhydrous ethanol alternately 3-5 times. After drying, obtain spherical iron oxide nanoparticles.

（1）将二价铁盐和三价铁盐溶解于去离子水中，配制成浓度为 0.15-1.0mol/L的溶液，在该溶液中加入固体NaOH，将pH值调节至10-12，室 温下搅拌10-60分钟，制得四氧化三铁黑色沉淀，过滤，用去离子水和无水乙 醇交替清洗3-5次，干燥后得到球状四氧化三铁纳米粒子；

[0011]

(2) Dissolve ferrous salts and ferric salts in deionized water to prepare a solution with a concentration of 0.15-1.0 mol/L. Then add sodium dodecyl sulfate to the solution, wherein the weight ratio of sodium dodecyl sulfate to water in the solution is 0.2-0.5:100 to obtain a mixed solution. Add solid NaOH to the mixed solution to adjust the pH value to 10-12 to obtain a reaction solution. Place the reaction solution under a visible light source with a wavelength of 400-750 nm for 10-60 minutes while stirring to obtain a black precipitate of iron oxide. Filter

the precipitate and wash it alternately with deionized water and anhydrous ethanol 3-5 times. After drying, cubic iron oxide nanoparticles are obtained.

（2）将二价铁盐和三价铁盐溶解于去离子水中，配制成浓度为 0.15-1.0mol/L的溶液，再向溶液中加入十二烷基硫酸钠，其中，十二烷基硫酸 钠与溶液中的水之间的重量比为0.2-0.5：100，得到混合溶液；在该混合溶液 中加入固体NaOH，将pH值调节至10-12，得到反应液；并将反应液放置在波长 为400-750nm的可见光光源下照射10-60分钟，同时搅拌，制得四氧化三铁黑 色沉淀，过滤，用去离子水和无水乙醇交替清洗3-5次，干燥后得到立方体状 的四氧化三铁纳米粒子。

[0012]

(3) Dissolve ferrous salts and ferric salts in deionized water to prepare a solution with a concentration of 0.15-1.0 mol/L. Then add sodium dodecyl sulfate to the solution, wherein the weight ratio of sodium dodecyl sulfate to water in the solution is 0.8-1.2:100 to obtain a mixed solution. Add solid NaOH to the mixed solution to adjust the pH value to 10-12 to obtain a reaction solution. Place the reaction solution under a visible light source with a wavelength of 400-750 nm for 10-60 minutes while stirring to obtain a black precipitate of iron tetroxide.

Filter the precipitate and wash it alternately with deionized water and anhydrous ethanol 3-5 times. After drying, rod-shaped iron tetroxide nanoparticles are obtained.

（3）将二价铁盐和三价铁盐溶解于去离子水中，配制成浓度为 0.15-1.0mol/L的溶液，再向溶液中加入十二烷基硫酸钠，其中，十二烷基硫酸 钠与溶液中的水之间的重量比为0.8-1.2：100，得到混合溶液；在该混合溶液 中加入固体NaOH，将pH值调节至10-12，得到反应液；并将反应液放置在波长 为400-750nm的可见光光源下照射10-60分钟，同时搅拌，制得四氧化三铁黑 色沉淀，过滤，用去离子水和无水乙醇交替清洗3-5次，干燥后得到棒状四氧 化三铁纳米粒子。

[0013]

The iron salts mentioned above include one of sulfates, nitrates, and chlorides.

如上所述的铁盐包括硫酸盐、硝酸盐和氯化盐等中的一种。

[0014]

The molar ratio of Fe²⁺ to Fe³⁺ in the divalent and trivalent ferric salts is 1:1.7 to 2.0.

所述的二价铁盐和三价铁盐中Fe ²⁺ 与Fe ³⁺ 的摩尔比为1：1.7～2.0。

[0015]

Compared with the prior art, the present invention has the following advantages:

与现有技术相比，本发明具有以下优点：

[0016]

(1) Using iron salts as raw materials, spherical, cubic and rod-shaped iron oxide nanoparticles can be prepared simply, quickly and effectively by co-precipitation method.

（1）以铁盐为原料，采用共沉淀法简单、快速、有效地制备得到球状、立 方体状、棒状的四氧化三铁纳米粒子。

[0017]

(2) Fe ₃ O ₄ nanospheres were prepared without the addition of sodium dodecyl sulfate and without visible light irradiation.

（2）在不添加十二烷基硫酸钠，不进行可见光照射的条件下，制备得到了 Fe ₃ O ₄ 纳米球。

Fe₃O₄ nanocubes and nanorods can be easily prepared by adjusting the amount of sodium dodecyl sulfate added and the irradiation time with visible light.

通过调节十二烷基硫酸钠的添加量和可见光的照射时间，可方便 地制得Fe ₃ O ₄ 纳米立方体和纳米棒。

[0018]

(3) The prepared iron oxide nanoparticles have good dispersibility, uniform particle size, small particle size, high purity, and high saturation magnetization. The morphology can be controlled to be spherical, cubic, or rod-shaped.

（3）制备得到的四氧化三铁纳米粒子分散性好、粒子大小均匀、粒径小、 纯度高、饱和磁化强度高，形貌可实现球状、立方状和棒状的调控。

[0019]

(4) The preparation method is relatively simple, the production cost is low, the morphology is easy to control, and it is easy to achieve large-scale industrial production.

（4）制备方法相对简单、生产成本低廉、形貌易调控，易于实现规模化的 工业生产。

[0020]

Attached Figure Description

附图说明

[0021]

Figure 1 is a transmission electron microscope (TEM) image of the iron oxide nanospheres prepared in Example 1.

图1是实施实例1制备的四氧化三铁纳米球的透射电镜（TEM）照片

[0022]

Figure 2 is a transmission electron microscope (TEM) image of the iron oxide nanospheres prepared in Example 2.

图2是实施实例2制备的四氧化三铁纳米球的透射电镜（TEM）照片

[0023]

Figure 3 is a transmission electron microscope (TEM) image of the iron oxide nanospheres prepared in Example 3.

图3是实施实例3制备的四氧化三铁纳米球的透射电镜（TEM）照片

[0024]

Figure 4 is a transmission electron microscope (TEM) image of the iron oxide nanorods prepared in Example 4.

图4是实施实例4制备的四氧化三铁纳米棒的透射电镜（TEM）照片

[0025]

Figure 5 is a transmission electron microscope (TEM) image of the iron oxide nanorods prepared in Example 5.

图5是实施实例5制备的四氧化三铁纳米棒的透射电镜（TEM）照片

[0026]

Figure 6 shows the X-ray diffraction (XRD) pattern of the iron oxide nanorods prepared in Example 6.

图6是实施实例6制备的四氧化三铁纳米棒的X-射线衍射（XRD）图谱

[0027]

Figure 7 is a transmission electron microscope (TEM) image of the iron oxide nanocubes prepared in Example 7.

图7是实施实例7制备的四氧化三铁纳米立方体的透射电镜（TEM）照片

[0028]

Figure 8 is a transmission electron microscope (TEM) image of the iron oxide nanocubes prepared in Example 8.

图8是实施实例8制备的四氧化三铁纳米立方体的透射电镜（TEM）照片

[0029]

Figure 9 is a hysteresis loop diagram of the iron oxide nanocubes prepared in Example 8.

图9是实施实例8制备的四氧化三铁纳米立方体的磁滞回线图

[0030]

Figure 10 is a transmission electron microscope (TEM) image of the iron oxide nanocubes prepared in Example 9.

图10是实施实例9制备的四氧化三铁纳米立方体的透射电镜（TEM）照片。

[0031]

Detailed Implementation

具体实施方式

[0032]

Example 1

实施例1

[0033]

Weigh 1.4g of FeSO・7HO and 2.0g of Fe(SO)O and dissolve them in 100mL of deionized water. Stir thoroughly to completely dissolve the ferric sulfate and ferrous sulfate to obtain a solution with a concentration of 0.15mol/L. Add solid NaOH to the solution and dissolve it completely. Adjust the pH to 10 and stir at room temperature for 30 minutes to obtain a black precipitate of iron(III) oxide. After filtration, wash the precipitate three times with deionized water and anhydrous ethanol, respectively. Dry at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取1.4g FeSO ₄ ・7H ₂ O和2.0g Fe ₂ (SO <sub> 4 < /sub>) ₃ 溶于100mL去离子水中，充分搅拌 使硫酸铁和硫酸亚铁完全溶解，得到浓度为0.15mol/L的溶液，将固体NaOH加 入到该溶液中，充分溶解后使pH值调节至10，室温下搅拌30分钟，得到四氧 化三铁黑色沉淀物，过滤后用去离子水和无水乙醇分别清洗沉淀物3次，室温 干燥24小时，得到四氧化三铁纳米粒子。

The transmission electron microscopy observation results are shown in Figure 1. It can be

seen that the iron oxide nanoparticles are spherical with a particle size distribution between 5-20 nm. The particles are uniform in size and there is no aggregation.

透射电镜观察结果如图1所示，可以 看到，四氧化三铁纳米粒子呈球形，粒径分布在5-20nm之间，粒子大小均匀， 无团聚现象。

[0034]

Example 2

实施例2

[0035]

Weigh 7.36g of FeClO and 17.0g of FeClO and 6HO, respectively, and dissolve them in 100mL of deionized water. Stir thoroughly to completely dissolve the iron salts, obtaining a solution with a concentration of 1.0mol/L. Add solid NaOH to this solution and dissolve it completely. Adjust the pH to 12 and stir at room temperature for 60 minutes to obtain a black precipitate of iron(III) oxide. After filtration, wash the precipitate five times with deionized water and five times with anhydrous ethanol, respectively. Dry at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取7.36g FeCl ₂ ・4H ₂ O和17.0g FeCl ₃ ・6H ₂ O溶于100mL去离子水中，充 分搅拌使铁盐完全溶解，得到浓度为1.0mol/L的溶液，将固体NaOH加入到该 溶液中，充分溶解后使pH值调节至12，室温下搅拌60分钟，得到四氧化三铁 黑色沉淀物，过滤后用去离子水和无水乙醇分别清洗沉淀物5次，室温干燥24 小时，得到四氧化三铁纳米粒子。

The transmission electron microscopy observation results are shown in Figure 2. It can be seen that the iron oxide nanoparticles are spherical with a particle size distribution between 10-15 nm. The particles are uniform in size and there is no aggregation.

透射电镜观察结果如图2所示，可以看到， 四氧化三铁纳米粒子呈球形，粒径分布在10-15nm之间，粒子大小均匀，无团 聚现象。

[0036]

Example 3

实施例3

[0037]

Weigh 4.3g of Fe(NO3-NER22-)-NER23-・6H24-O and 12.1g of Fe(NO3-NER25-)-NER26-・9H27-O and dissolve them in 100mL of deionized water. Stir thoroughly to completely dissolve the iron salts, obtaining a solution with a concentration of 0.45mol/L. Add solid NaOH to this

solution and dissolve it completely. Adjust the pH value to 11 and stir at room temperature for 10 minutes to obtain a black precipitate of iron(III) oxide. After filtration, wash the precipitate four times with deionized water and four times with anhydrous ethanol, and dry it at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取4.3g Fe(NO ₃) ₂ ・6H ₂ O和12.1g Fe(NO ₃) ₃ ・9H ₂ O溶于100mL去离子水中， 充分搅拌使铁盐完全溶解，得到浓度为0.45mol/L的溶液，将固体NaOH加入到 该溶液中，充分溶解后使pH值调节至11，室温下搅拌10分钟，得到四氧化三 铁黑色沉淀物，过滤后用去离子水和无水乙醇分别清洗沉淀物4次，室温干燥 24小时，得到四氧化三铁纳米粒子。

The transmission electron microscopy observation results are shown in Figure 3. It can be seen that the iron oxide nanoparticles are spherical with a particle size distribution between 5-15 nm. The particles are uniform in size and there is no aggregation.

透射电镜观察结果如图3所示，可以看到， 四氧化三铁纳米粒子呈球形，粒径分布在5-15nm之间，粒子大小均匀，无团聚 现象。

[0038]

Example 4

实施例4

[0039]

Weigh 1.0 g of FeClO and 2.7 g of FeClO and 6 HO, respectively, and dissolve them in 100 mL of deionized water to obtain a solution with a concentration of 0.15 mol/L. Add 0.2 g of sodium dodecyl sulfate to this solution and stir thoroughly until the sodium dodecyl sulfate is completely dissolved to obtain a mixed solution. Add solid NaOH to this mixed solution to adjust the pH to 11 to obtain a reaction solution. Place the reaction solution under a visible light source with a wavelength of 400 nm for 10 minutes and stir continuously with magnetic force during irradiation to obtain a black precipitate of iron(III) oxide. After filtration, wash the

precipitate three times with deionized water and anhydrous ethanol, respectively, and dry it at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取1.0gFeCl ₂ ·4H ₂ O和2.7gFeCl ₃ ·6H <sub> 2 < /sub> O溶于100mL去离子水中，得到浓 度为0.15mol/L的溶液，在该溶液中加入0.2g十二烷基硫酸钠，充分搅拌，使 十二烷基硫酸钠完全溶解，得到混合溶液；将固体NaOH加入到此混合溶液中，使pH值调节至11，得到反应液，并将反应液放置在波长为400nm的可见光光源 下照射10分钟，照射期间持续磁力搅拌，制得四氧化三铁黑色沉淀物，过滤后 用去离子水和无水乙醇分别清洗沉淀物3次，室温干燥24小时，得到四氧化三 铁纳米粒子。

The transmission electron microscopy observation results are shown in Figure 4. It can be seen that the iron oxide nanoparticles have a rod-shaped morphology, with a length of 100±30 nm and a width of 12±3 nm. The particles have good uniformity in size and are monodisperse.

透射电镜观察结果如图4所示，可以看到，四氧化三铁纳米粒子 形貌为棒状，粒子的长度为100±30nm，宽度为12±3nm，粒子大小均匀性好， 呈单分散性。

[0040]

Example 5

实施例5

[0041]

Weigh 5.76 g of Fe(NO3-NER32-)-NER33-・6H-NER34-O and 16.16 g of Fe(NO3-NER35-)-NER36-・9H-NER37-O and dissolve them in 100 mL of deionized water to obtain a solution with a concentration of 0.6 mol/L. Add 0.3 g of sodium dodecyl sulfate to this solution and stir thoroughly until the sodium dodecyl sulfate is completely dissolved to obtain a mixed solution. Add solid NaOH to this mixed solution to adjust the pH value to 10 to obtain a reaction solution. Place the reaction solution under a visible light source with a wavelength of 600 nm for 30 minutes and stir continuously with magnetic force during irradiation to obtain a black precipitate of iron(III) oxide. After filtration, wash the precipitate four times with

deionized water and anhydrous ethanol, respectively, and dry it at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取5.76g Fe(NO ₃) ₂ ·6H ₂ O和16.16g Fe(NO ₃) ₃ ·9H ₂ O溶于100mL去离子水 中，得到浓度为0.6mol/L的溶液，在该溶液中加入0.3g十二烷基硫酸钠，充 分搅拌，使十二烷基硫酸钠完全溶解，得到混合溶液；将固体NaOH加入到此混 合溶液中，使pH值调节至10，得到反应液，并将反应液放置在波长为600nm的 可见光光源下照射30分钟，照射期间持续磁力搅拌，制得四氧化三铁黑色沉淀 物，过滤后用去离子水和无水乙醇分别清洗沉淀物4次，室温干燥24小时，得 到四氧化三铁纳米粒子。

The transmission electron microscopy (TEM) results are shown in Figure 5. It can be seen that the iron oxide nanoparticles have a rod-like morphology, with a length of 100±20 nm and a width of 12±2 nm. The particles are uniform in size and have good dispersibility.

透射电镜观察结果如图5所示，可以看到，四氧化三 铁纳米粒子形貌为棒状，粒子的长度为100±20nm，宽度为12±2nm，粒子大小 均匀，分散性好。

[0042]

Example 6

实施例6

[0043]

Weigh 9.2g of FeSO・7HO and 13.2g of FeSO(SO) and dissolve them in 100mL of deionized water to obtain a solution with a concentration of 1.0mol/L. Add 0.5g of sodium dodecyl sulfate to this solution and stir thoroughly until the sodium dodecyl sulfate is completely dissolved to obtain a mixed solution. Add solid NaOH to this mixed solution to adjust the pH to 12 to obtain a reaction solution. Place the reaction solution under a visible light source with a wavelength of 750nm for 60 minutes and stir continuously with magnetic force during irradiation to obtain a black precipitate of iron(III) oxide. After filtration, wash the precipitate five times with deionized water and anhydrous ethanol, respectively, and dry it at room temperature for 24 hours to obtain rod-shaped iron(III) oxide nanoparticles.

称取9.2g FeSO ₄ ·7H ₂ O和13.2g Fe ₂ (SO <sub> 4 < /sub>) ₃ 溶于100mL去离子水中，得到浓 度为1.0mol/L的溶液，在该溶液中加入0.5 g十二烷基硫酸钠，充分搅拌，使 十二烷基硫酸钠完全溶解，得到混合溶液；将固体NaOH加入到此混合溶液中， 使pH值调节至12，得到反应液，并将反应液放置在波长为750nm的可见光光源 下照射60分钟，照射期间持续磁力搅拌，制得四氧化三铁黑色沉淀物，过滤后 用去离子水和无水乙醇分别清洗沉淀物5次，室温干燥24小时，得到棒状的四 氧化三铁纳米粒子。

The particles have a length of 100±15nm and a width of 10±2nm, exhibiting uniform particle size and good dispersion.

粒子的长度为100±15nm，宽度为10±2nm，粒子大小均匀， 分散性好。

The X-ray diffraction analysis results are shown in Figure 6. It can be seen that the product exhibits obvious Fe ₃ O ₄ characteristic diffraction peaks with high intensity and no other impurity peaks, indicating that the prepared rod-shaped iron tetroxide nanoparticles have high crystallinity and purity.

X-射线衍射分析结果如图6所示，可以看到，产物表现出明显的Fe ₃ O <sub> 4 < /sub> 特征衍射峰，衍射峰强度很高，且无其它杂峰，说明制备得到的棒状四氧化三 铁纳米粒子具有较高的结晶度和纯度。

[0044]

Example 7

实施例7

[0045]

Weigh 1.44g of Fe(NO3-NER45)3-NER46-・6H2O and 4.0g of Fe(NO3-NER48)3-NER49-・9H2O and dissolve them in 100mL of deionized water. Stir thoroughly to completely dissolve ferrous

chloride and ferric chloride to obtain a solution with a concentration of 0.15mol/L. Add 0.8g of sodium dodecyl sulfate to this solution and stir thoroughly to completely dissolve the sodium dodecyl sulfate to obtain a mixed solution. Add solid NaOH to this mixed solution to adjust the pH to 10 to obtain a reaction solution. Place the reaction solution under a visible light source with a wavelength of 750nm for 10 minutes and stir magnetically continuously during irradiation to obtain a black precipitate of iron(III) oxide. After filtration, wash the precipitate three times with deionized water and anhydrous ethanol, respectively, and dry at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取1.44g Fe(NO ₃) ₂ ·6H ₂ O和4.0g Fe(NO ₃) ₃ ·9H ₂ O溶于100mL去离子水中， 充分搅拌使氯化亚铁和氯化铁完全溶解，得到浓度为0.15mol/L的溶液，在该 溶液中加入0.8g十二烷基硫酸钠，充分搅拌，使十二烷基硫酸钠完全溶解，得 到混合溶液；将固体NaOH加入到此混合溶液中，使pH值调节至10，得到反应 液，并将反应液放置在波长为750nm的可见光光源下照射10分钟，照射期间持 续磁力搅拌，得到四氧化三铁黑色沉淀物，过滤后用去离子水和无水乙醇分别 清洗沉淀物3次，室温干燥24小时，得到四氧化三铁纳米粒子。

The results observed by transmission electron microscopy are shown in Figure 7. As can be seen from Figure 7, the iron oxide nanoparticles exhibit a regular cubic shape with a side

length of 5-15 nm. The particle size distribution range is narrow, the size is uniform, and they are monodisperse.

透射电镜观察 结果如图7所示，从图7可以看出，四氧化三铁纳米粒子呈现出规则的立方体 状，立方体的边长为5-15nm，粒径分布范围窄，大小均匀，呈单分散性。

[0046]

Example 8

实施例8

[0047]

Weigh 3.0 g of FeClO and 7.3 g of FeClO and 6 HO, respectively, and dissolve them in 100 mL of deionized water. Stir thoroughly to completely dissolve ferrous chloride and ferric chloride, obtaining a solution with a concentration of 0.42 mol/L. Add 1.0 g of sodium dodecyl sulfate to this solution and stir thoroughly to completely dissolve the sodium dodecyl sulfate, obtaining a mixed solution. Add solid NaOH to this mixed solution to adjust the pH to 12, obtaining a reaction solution. Place the reaction solution under a visible light source with a wavelength of 500 nm for 40 minutes, and continuously stir magnetically during irradiation to obtain a black precipitate of iron(III) oxide. After filtration, wash the precipitate four times with deionized water and anhydrous ethanol, respectively, and dry at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取3.0g FeCl ₂ ·4H ₂ O和7.3g FeCl ₃ ·6H <sub> 2 < /sub> O溶于100mL去离子水中，充分 搅拌使氯化亚铁和氯化铁完全溶解，得到浓度为0.42mol/L的溶液，在该溶液 中加入1.0g十二烷基硫酸钠，充分搅拌，使十二烷基硫酸钠完全溶解，得到混 合溶液；将固体NaOH加入到此混合溶液中，使pH值调节至12，得到反应液， 并将反应液放置在波长为500nm的可见光光源下照射40分钟，照射期间持续磁 力搅拌，得到四氧化三铁黑色沉淀物，过滤后用去离子水和无水乙醇分别清洗 沉淀物4次，室温干燥24小时，得到四氧化三铁纳米粒子。

The results of transmission electron microscopy observation and magnetic property testing are shown in Figures 8 and 9, respectively.

透射电镜观察和磁 性能测试结果分别如图8和9所示。

As can be seen from Figure 8, the iron oxide nanoparticles exhibit a regular cubic shape with a side length of 15-25 nm, uniform particle size, and monodispersity.

从图8可以看出，四氧化三铁纳米粒子呈 现出规则的立方体状，立方体的边长为15-25nm，粒径大小均匀，呈单分散性。

Figure 9 shows the hysteresis loop of the Fe3O4 nanocube. The saturation magnetization of the product is 91.3 emu/g, which is close to the saturation magnetization of bulk Fe₃ O₄ (~90 emu/g), indicating that the product has high magnetic properties.

图9给出了四氧化三铁纳米立方体的磁滞回线，产物的饱和磁化强度为 91.3emu/g，与块状Fe ₃ O ₄ 的饱和磁化强度接近（～90emu/g），说明产物具有较高 的磁性能。

[0048]

Example 9

实施例9

[0049]

Weigh 9.2g of FeSO・7HO and 13.2g of FeSO(SO)NER61 and dissolve them in 100mL of deionized water to prepare a 1.0mol/L solution. Add 1.2g of sodium dodecyl sulfate to this solution and stir thoroughly until the sodium dodecyl sulfate is completely dissolved. Then add solid NaOH to this solution and adjust the pH to 11 to obtain a reaction solution. Place the reaction solution under a visible light source with a wavelength of 400nm for 60 minutes and

stir continuously with a magnetic force during the irradiation to obtain a black precipitate of iron(III) oxide. Finally, wash the precipitate five times with deionized water and anhydrous ethanol, respectively, and dry it at room temperature for 24 hours to obtain iron(III) oxide nanoparticles.

称取9.2g FeSO ₄ · 7H ₂ O和13.2g Fe ₂ (SO ₄) ₃ 溶于100mL去离子水中，配制成 浓度为1.0mol/L的溶液，在该溶液中加入1.2g十二烷基硫酸钠，充分搅拌， 使十二烷基硫酸钠完全溶解，然后将固体NaOH加入到此溶液中，调节pH值至 11，得到反应液，并将反应液放置在波长为400nm的可见光光源下照射60分钟， 照射期间持续磁力搅拌，得到四氧化三铁黑色沉淀物，最后用去离子水和无水 乙醇分别清洗沉淀物5次，室温干燥24小时，得到四氧化三铁纳米粒子。

The transmission electron microscopy observation results are shown in Figure 10. It can be seen that the iron oxide nanoparticles exhibit a regular cubic shape with a side length of 5-13 nm. The particle size distribution range is narrow, the size is uniform, and the dispersion is good.

透射 电镜观察结果如图10所示，可以看出，四氧化三铁纳米粒子呈现出规则的立方 体状，立方体的边长分布在5-13nm，粒径分布范围窄，大小均匀，分散性好。