

**HANOI UNIVERSITY OF SCIENCE AND TECHNOLOGY
INTERNATIONAL TRAINING INSTITUTE FOR MATERIALS SCIENCE**

**PROCEEDINGS
ICAMN 2022**

**THE 5TH INTERNATIONAL CONFERENCE ON ADVANCED
MATERIALS AND NANOTECHNOLOGY**

NOVEMBER 16TH – 19TH, 2022

BACH KHOA PUBLISHING HOUSE

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Study on the manufacture and application of nano Fe₃O₄ materials doped with manganese for treatment of water contaminated with Arsenic (III)

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Abstract: This article reports results of a research on manufacture and application of Fe₃O₄ nanomaterial doped with Mn (Fe₃O₄-x%Mn) for the treatment of water contaminated with As(III). The materials were synthesized by chemical method and its particle size was studied by the XRD and TEM techniques. Experimental results showed that the materials were in crystallized form of cubic face centered structure with particles sizes ranging from 8 to 12 nm. The materials have paramagnetic property with values of magnetization (Ms) of 53 ÷ 65.8 emu/g at room temperature. The best adsorption property of the materials towards As was observed in neutral aquatic solutions.

Keywords: Arsenic adsorption, Fe₃O₄ nanoparticles doped with Mn, Mangan, TEM technique, XRD technique.

I. INTRODUCTION

The transition metal-doped Fe₃O₄ magnetic nanomaterials are researched and manufactured because they are applied in many fields such as information storage, printing industry, soft magnetic magnets, cancer treatment, magnetic ultrasound, environmental treatment, etc [1-5]. Nanoparticles exhibit special properties because the ratio of their surface area to mass increases [1-3]. Nanoparticles exhibit special properties because the ratio of their surface area to mass increases [1-3]. As the ratio between surface area and particle mass increases, the electrical and magnetic properties change significantly due to the particle size quantum effect. At the same time, the particles tend to move to the minimum free energy levels through a number of forms such as phase transition, germinating, surface structure change [3, 4]. The important property of Fe₃O₄ iron oxide nanomaterials is that it is non-toxic to the environment and easy to separate adsorbed substances from the material. Therefore, scientists have been interested in research to improve the As adsorption capacity of iron oxide nanomaterials from Fe₃O₄ in aqueous environment [5, 6, 9, 10]. However, the publications have not fully mentioned the influence of Mn transition metal doping on the microstructure and magnetism of Fe₃O₄ ferromagnetic nanomaterials as well as the applicability of the material to treat As in the aquatic environment.

This paper reports the results of studying the microstructure, magnetic properties of Mn-doped

Fe₃O₄ magnetic iron oxide nanomaterials and investigating the adsorption capacity of As in water with different pH.

II. EXPERIMENTAL

A. Chemistry

The chemicals used are FeCl₃.6H₂O, purity 99%; Na₂SO₃ purity 99%; (CH₃COO)₂Mn.4H₂O purity 99% (Merck), acetone purity 99% (Merck). NH₃ solution 25%, As₂O₃ stock solution made from As₂O₃ with a concentration of 1 g/l (1g/l = 106 ppb, 105 times higher than the allowable As content) in domestic water [7]. Solution of NaOH (50%, purity 99%, Merck) and HCl (32%, purity 99%, Merck) were mixed in deionized water to adjust the pH in the study of As adsorption capacity of the materials.

B. Tool

A pH meter (TOA - Japan) is used to measure the pH of an aqueous solution containing As. The crystalline properties and grain sizes of the materials were studied by X-ray diffraction (XRD) and transmission electron microscopy (TEM) imaging techniques using the D5005 respectively (Bruker, Germany) and the JEOL TEM 5410NV instrument (Bruker, Germany). XRD and TEM analysis were conducted at Analytical Chemistry Laboratory, Institute of Chemistry, Vietnam Academy of Science and Technology.

The magnetism of the materials was determined by a vibrating sample magnetometer (VSMVibrating Sample Magnetometer) on the MICROSENE EV11 (Japan).

The concentration of As in the solution was quantified by atomic absorption method using AAS Shimadzu 630 instrument of the Institute of Nuclear Science and Technology, Vietnam Institute of Atomic Energy.

C. Experiment

Mix the Fe³⁺ solution into the Mn²⁺ solution so that the doping ratio is 0, 5, 10, 15, 20 and 30%. Pour the mixture of Fe³⁺ and Mn²⁺ solutions with different doping ratios into 200 ml of Na₂SO₃ solution and stir until the solution turns yellow. Then, slowly add NH₃ solution until pH = 10, stir the solution for 30 minutes, get a black solution. Use magnets to deposit particles of magnetic material. Then decant the solution. Incubated at 50°C under low pressure (less than 0.1 atm) for 2 days, obtained nanomaterials from Fe₃O₄-Mn with different Mn doping concentrations, denoted as Fe₃O₄, Fe₃O₄-5 %Mn, Fe₃O₄-10%Mn, Fe₃O₄-15%Mn, Fe₃O₄-20%Mn and Fe₃O₄-30%Mn. Put a certain amount of material with the above-mentioned Mn doped content into an aqueous solution containing As(III) with a concentration 10 times higher than the allowable level for domestic water and stir the solution for 20 minutes at room temperature. Previous experiments showed that with 20 minutes of stirring, As adsorption on the material will reach equilibrium. Use a magnet to separate the material from the solution. Quantitative As concentration in the solution after adsorption to determine the As adsorption capacity of the material.

III. RESULTS AND DISCUSSION

A. Microstructure of Mn doped Fe₃O₄ nanostructures

Figure 1 presents the X-ray diffraction spectrum of Fe₃O₄ and Fe₃O₄-5% Mn samples. From Figure 1, it can be seen that the diffraction peaks of the material coincide with the standard spectrum of the NaCl lattice showing that the samples of the material are single-phase and have a face-centered cubic structure. The lattice constant *d* of the Fe₃O₄ nanosample is calculated according to Bragg's formula:

$$2d\sin\theta = n\lambda \quad (1)$$

In which θ is the diffraction angle (also known as the Bragg angle), n is an integer, and λ is the wavelength of the X-ray beam hitting the sample.

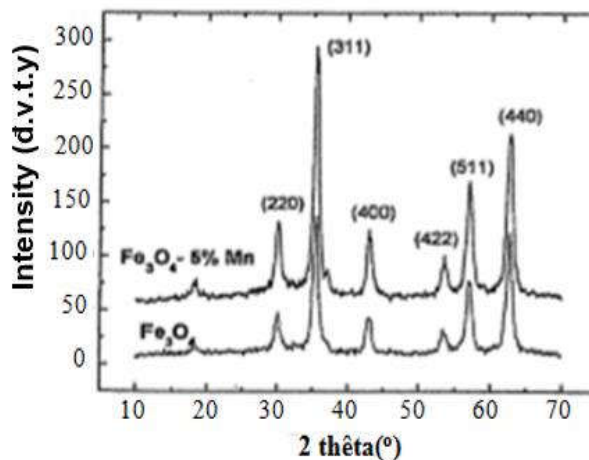


Figure 1. X-ray diffraction spectrum of Fe₃O₄ and Fe₃O₄-5%Mn samples compared with standard diffraction spectrum of NaCl crystal lattice with face-centered cubic structure.

The lattice constant value *d* was determined to be 0.8389 nm, quite similar to the standard lattice constant of NaCl crystals of 0.8396 nm.

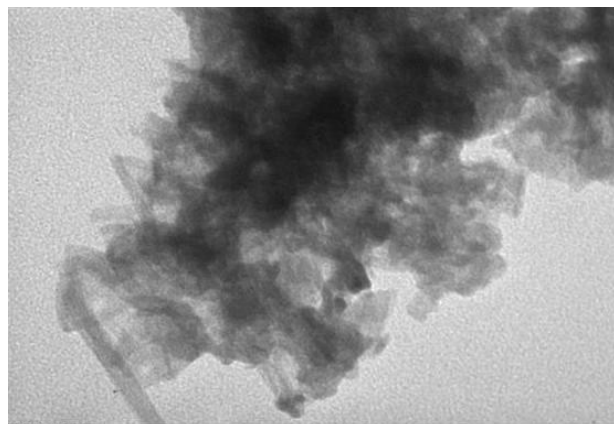
The size of the material crystals is calculated using the Debye - Scherrer formula:

$$D = \frac{0,9\lambda}{\beta \cos\theta} \quad (2)$$

In which β is the width at half height of the intensity peak (FWHM) of the diffracted beam in the XRD analysis, in radians. The symbols λ and θ are the same as in expression (1).

Experimental results show that the Fe₃O₄ crystals have a size of $D = 8.8$ nm.

Figure 2 is a TEM image of the Fe₃O₄ sample showing the clustering of particles. However, many particles with sizes between 8.4 and 12.7 nm can be seen. Similar to the Fe₃O₄ sample, the XRD analysis results (Figure 1) allow the lattice constant (*d*) of the Fe₃O₄-5%Mn sample to be calculated as 0.8377 nm. Compared with the lattice constant of Fe₃O₄ crystals, the *d*-value of Fe₃O₄-5%Mn lattice is narrower, but the difference is very small. This is explained because the Mn²⁺ ion radius is smaller than the Fe²⁺ ion radius, so when the Mn²⁺ ion replaces the Fe²⁺ ion in the lattice, the distance between the crystal faces narrows.



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Figure 2. TEM image of the Fe₃O₄ nanocrystal sample

B. Magnetic properties of samples

Figure 3 presents the magnetic moment (Ms) of ferromagnetic oxide nanomaterials with different Mn doped content. From Figure 3, it can be seen that, at room temperature, Fe₃O₄, Fe₃O₄-5%Mn, Fe₃O₄-10%Mn, Fe₃O₄-15%Mn, Fe₃O₄-20%Mn and Fe₃O₄-30%Mn materials exhibit superparamagnetic properties with the saturation magnetic moment is about 65.8 emu/g. This is evidence that Mn has replaced Fe in the lattice of the material because Mn has paramagnetic properties.

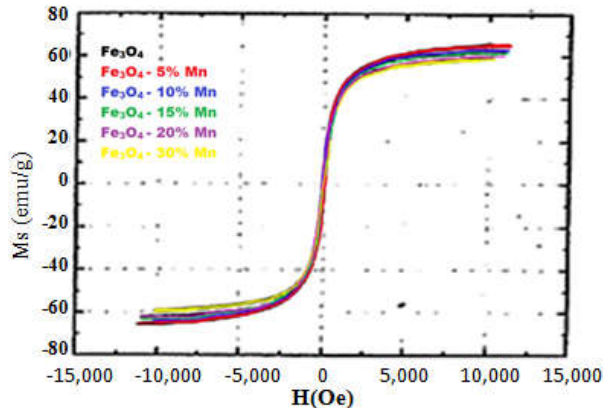


Figure 3. Magnetic moment (Ms) of ferromagnetic oxide nano samples with different Mn doped content

The saturation magnetic moment values of Fe₃O₄ and Fe₃O₄ in Mn phase with different contents are presented in Table 1. The results presented in Table 1 show that the saturation magnetic moment of the material decreases as the Mn impurity concentration increases, but the fluctuation of magnetic moment in the range of Mn doping content of 20-30% is not significant.

The decrease in the saturation magnetic moment when the Mn content in the material increases is explained by the fact that Mn has a weaker magnetism than Fe, so when Mn²⁺ replaces Fe²⁺ in the crystal lattice, the magnetism of the material will decrease.

Table 1. Magnetic saturation moment (Ms) of Mn doped Fe₃O₄ and Fe₃O₄ samples

Sample	Ms, emu/g
Fe ₃ O ₄	65.8
Fe ₃ O ₄ -5%Mn	65.3
Fe ₃ O ₄ -10%Mn	62.9
Fe ₃ O ₄ -15%Mn	61.9
Fe ₃ O ₄ -20%Mn	59.3
Fe ₃ O ₄ -30%Mn	59.5

C. Arsen adsorption capacity of Fe₃O₄-x%Mn nanomaterials (x = 0, 5, 10, 15)

Table 2 presents the As adsorption capacity in water of Mn-doped Fe₃O₄ and Fe₃O₄ nanomaterials with different concentrations. In Table 2, columns from No. 2 to No. 5 are the concentration of As remaining in the solution after stirring time for 20 minutes with different amounts (column 1) and type of adsorbent.

Table 2. Test results of As adsorption capacity in water of Fe₃O₄-x%Mn nanomaterials after 20 minutes of adsorption time at room temperature

Amount of adsorption (g/l)	Concentration of As remaining in solution (ppb). Material: Fe ₃ O ₄	Concentration of As remaining in solution (ppb). Material: Fe ₃ O ₄ -5%Mn	Concentration of As remaining in solution (ppb). Material: Fe ₃ O ₄ -10%Mn	Concentration of As remaining in solution (ppb). Material: Fe ₃ O ₄ -15%Mn
0	113	113	113	113
0.6	30.3460	31.6188	27.0847	37.0277
0.8	19.0508	27.2011	21.3576	29.5506
1.0	17.1020	24.2212	22.1530	26.1700
1.2	12.0111	21.9144	16.7012	25.6530
1.4	8.5510	16.9429	14.1588	13.4827
1.6	8.1135	17.1020	9.1078	17.7648
1.8	6.2417	9.5453	12.5680	15.6702
2.0	8.5510	5.1204	9.2271	12.8464
2.2	6.5823	9.5453	7.5965	16.1872

The results of Table 2 show that, in the case of ferromagnetic oxide nanomaterials with 10% Mn replacing iron in the crystal lattice and the amount of material (adsorbent) is 1.6 g/l, after 20 minutes of high concentration As level in water has decreased from 113 ppb, which is 10 times higher than the allowable standard [7], to below the standard level of 10 ppb (Table 2). The more Mn-doped samples have the higher the impurity content, the lower the As adsorption capacity. Fe₃O₄-5%Mn and Fe₃O₄-10%Mn materials showed the best adsorption: with the amount of 2 g/l, this material was able to reduce the concentration of As in water from 113 ppb to less than 10 ppb, ie reduce tens of times compared to the original concentration.

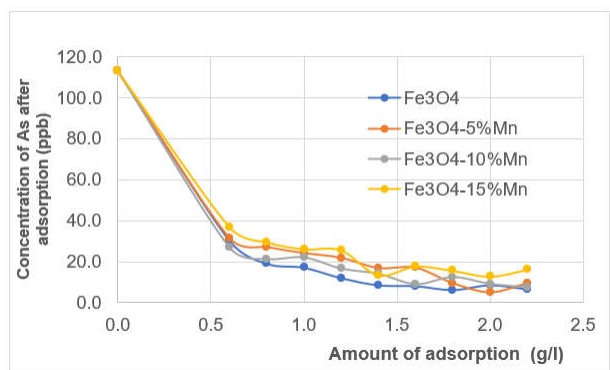


Figure 4. Effect of manganese (Mn) on arsenic (As) adsorption.

The test results show that the material has a good ability to adsorption As in water with a pH from neutral to weakly alkaline (pH = 5-8). In alkaline environment, pH > 10, the material exhibits poor adsorption for As.

IV. CONCLUSION

Fe₃O₄-x%Mn nanomaterials (x = 0; 5; 10; 15; 20 and 30) were synthesized by chemical methods and studied the structure and magnetic properties by XRD, TEM analysis techniques. The studied materials have particle sizes ranging from 8.42 to 12.7 nm, exhibit superparamagnetic properties at room temperature and have a saturation magnetic moment in the range of 53 ÷ 65.8 emu/g. Materials with < 10% Mn content replacing Fe in the crystal lattice have the ability to reduce As(III) concentration in water from a high level of hundreds of ppb to a level lower than 10 ppb, if the pH of the medium is neutral.

The limitation of this study is that the adsorption mechanism has not been determined as

well as thermodynamic parameters such as maximum adsorption capacity, adsorption equilibrium constant, Gibb free energy (ΔG^0), enthalpy (ΔH^0), entropy (ΔS^0) of the As adsorption process in water of the material. These studies will be implemented in the near future.

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PROCEEDINGS ICAMN 2022
**The 5th International Conference on Advanced Materials
and Nanotechnology**

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Responsible for Publication:

Director – Editor-in-Chief: Dr. Bui Duc Hung

Editor: Do Thanh Thuy

Book designer: Vu Thi Hang, Nguyen Thi Thu

Cover designer: Phan Le Huy San

Printed at: Vietmax Investment and Development Joint Stock Company, D10-11, Tan Trieu, Thanh Tri, Hanoi.

Quantity: 100 copies. Size: (21 × 29.7) cm.

Registered number: 3915-2022/CXBIPH/03-76/BKHN; ISBN: 978-604-316-915-7.

Publishing decision number: 198/QĐ-ĐHBK-BKHN, 08/11/2022.

Archived: IV – 2022.