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Chemically Synthesis and Characterization of Iron and Nickel Oxide Nanoparticles for Biomedicine

Seyed Javad Davarpanah¹, Ramin Karimian^{1*}, Farideh Piri²

Abstract

Magnetite (Fe_3O_4 and NiO) nanoparticles with a size range of 40–60 nm were prepared by sol-gel technique in nano and micro reverse micelles (water- in-oil). The surface properties, size, morphology and crystallographic structure of Fe_3O_4 and NiO particles are characterized by means of X-ray diffraction, transmission electron microscope and scanning electron microscope which will give much valuable information about these materials. In addition, synthesis of nanoparticles can be easily implemented because it is simple and environmentally friendly.

1. Applied Biotechnology Research Center, Baqiyatallah University of Medical Sciences, Tehran, Iran

2. Department of Chemistry, Faculty of Science, Zanzan University, Zanzan, Iran

* Corresponding Author

Ramin Karimian

Applied Biotechnology Research Center, Baqiyatallah University of Medical Sciences, Tehran, Iran

E-mail: karimian.r@gmail.com

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Introduction

Biocompatible materials exhibiting response to external magnetic field have found many interesting applications in various areas of biosciences and biotechnology, including different medical disciplines. The broad family of magnetic field controllable materials includes both nano and microparticles, high aspect ratio structures (nanotubes, nanowires), thin films, etc. Ferrofluids (magnetic fluids), magnetorheological fluids, magnetic polymers, magnetic inorganic materials, magnetically modified biological structures, magnetic particles with bound biomolecules etc. can serve as typical examples. In many cases magnetically responsive composite materials consist of small magnetic particles (most often formed by magnetite, maghemite or various ferrites), usually in the nanometer to micrometer range, dispersed in a polymer, biopolymer or inorganic matrix; alternatively magnetic particles can be adsorbed on the outer surface of diamagnetic particles [1-3].

In most cases synthetic (laboratory produced) magnetically responsive nano and microparticles and related structures have been developed, however, biologically produced magnetic particles (e.g., magnetosomes produced by magnetotactic bacteria) have been successfully used for selected bioapplications [4]. This study a typical example of biocompatible magnetic materials synthesis and typical examples of their biomedical applications. Magnetic nanoparticles have found various applications in ferrofluids, targeted drug delivery, magnetic resonance imaging (MRI), RNA and DNA purification, immunoassays, enzyme immobilization, hyperthermia treatment and catalysis [5-7]. Researchers have developed

several methods over the past decade to synthesize magnetic nanoparticles [8-10] and from these techniques the microemulsion method offers many advantages [11]. A microemulsion is an isotropic and thermodynamically stable single-phase liquid formed by at least three components: two immiscible components and a surfactant which is amphiphilic in nature. The water-in-oil microemulsion consists of reverse micelles with an aqueous core of nanometer size that serves as a nanoreactor in which the periphery formed by the surfactants physically confines the reaction. The water-in-oil microemulsion method has several advantages over other synthesis methods. Using this method, one can custom synthesize the particles to a given size and surface functionality. The water to surfactant ratio controls the size of the aqueous core, which in turn controls the size of the nanoparticles [12]. The micelles disperse the newly-formed nanoparticles in the oil phase lessening problems with subsequent agglomeration; and with proper chemical control, the resulting particles have a narrow size distribution [13]. The aqueous microenvironment within the nanoreactor allows the synthesis of nanoparticles with hydrophilic surfaces, particularly surfaces with extended hydroxyl (-OH) groups. These hydroxyl groups facilitate further surface functionalization [14]. In the present study, an original method for the synthesis of Iron (Fe_3O_4) and nickel oxide (NiO) nanoparticles by sol-gel technique in nano and micro micelles is described. The surface properties, size, morphology and crystallographic structure of Fe_3O_4 and NiO particles are characterized by means of X-ray diffraction (XRD), transmission electron microscope



(TEM) and scanning electron microscope (SEM) which will give much valuable information about these materials.

Materials and Methods

Chemicals and apparatus

All materials were commercial reagent grade and used without further purification. Chemical reagents ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, NaOH, Tween 80) and solvents were purchased from the Merck Chemical Company. Scanning electron microscope (SEM) was performed with a PHILIP XL-30, operated at 30 KV. Transmission electron microscopy (TEM) was performed with Zeiss EM10C-80 KV, operated at 80 KV. X-ray diffraction was conducted on a Philips Analytical XPERT diffractometer using a Cu K radiation ($\lambda = 1.54056 \text{ \AA}$) with a MINIPROP detector and operating at 40 mA and 40 kV. The purity determination of the substrates and reaction monitoring were accomplished by thin layer chromatography (TLC) on silica gel polygram SILG/UV 254 plates.

Preparation of magnetic oxide nanoparticles

Tween 80, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, NaOH, soybean oil and distilled water were used in the experiments. Nanoparticles were synthesized by the following steps: 0.60 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (or $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) in water (2 ml) and 4% Tween 80 were added into purified soybean oil (60 ml) under mechanical stirrer with 2200 rpm until obtaining a nearly clear emulsion. This solution was referred to as solution A. NaOH (0.35 g) was dissolved into water (2.5 ml) was added into solution A under mechanical stirrer with 2000 rpm for 2.5 h at room temperature and then the reaction mixture was filtrated. The precipitate was washed with absolute water ($3 \times 500 \text{ ml}$) for 3 times. This material was calcinated in electronic oven at 220°C for 3 h. The resulting powder X-ray diffraction was conducted on a Philips Analytical XPERT diffractometer using a Cu K radiation ($\lambda = 1.54056 \text{ \AA}$) with a MINIPROP detector and operating at 40 kV and 40 mA. X-ray diffraction patterns were recorded between $2\theta = 5^\circ$ and 79° with a step of 0.04° and a time of 0.8 s by step. The crystallographic data of the resulting Fe_3O_4 (or NiO) powders were collected by using the PC-APD, Diffraction software. Surface morphologies of the specimens were observed with a scanning electron microscope (SEM, Philips XL-30). The ordered nano structures of Fe_3O_4 (or NiO) can be further confirmed by transmission electron microscope (TEM, Philips KV-120).

Results

Scanning electron microscopy (SEM) and Transmission electron microscopy (TEM) images of Fe_3O_4 and NiO nanoparticles (Fig. 1, 2 & 3) indicating the homogeneous size, agglomeration of particles, with diameters ranging from 40 to 60 nm. The morphology of the prepared Fe_3O_4 and NiO nanoparticles was characterized by SEM and TEM images as shown in Figure 1-3. From images results, we can observe a large quantity of uniform nanoparticles (NPs) with average particle size of 40-60 nm, indicates that our synthesis process is an easy method for the preparation Fe_3O_4 and NiO nanoparticles. The synthesized magnetic nanoparticles oxide shows good nanoparticles

structure and are stable in hydrocarbon solvents against air oxidation. Figure 4 and 5 show the XRD patterns of Fe_3O_4 and NiO powders after calcinations at 200°C . The nano-sized Fe_3O_4 and NiO powders are in amorphous structure. Its average particle size was 48.51 nm for Fe_3O_4 and 41.23 nm for NiO nanoparticles. That calculated through the Scherrer's formula $D = 0.89 / B \cos \theta$. D , θ , B and λ were the average particle size, the X-ray wavelength, the angular line width of half maximum intensity and the Bragg's angle respectively. The phase composition and structure of obtained samples were examined by X-ray powder diffraction (XRD).

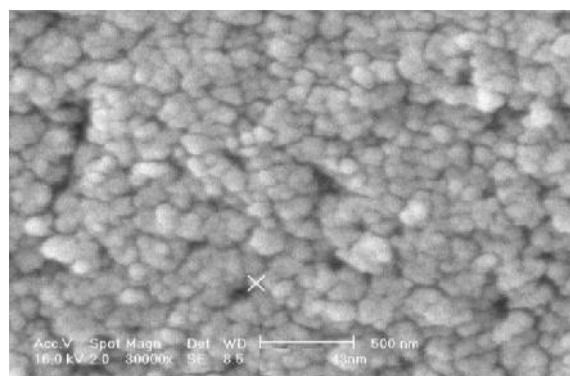


Figure 1. SEM image of the Fe_3O_4 nanoparticle

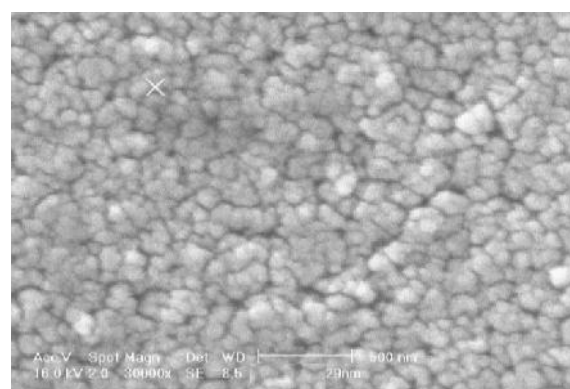


Figure 2. SEM image of the NiO nanoparticle

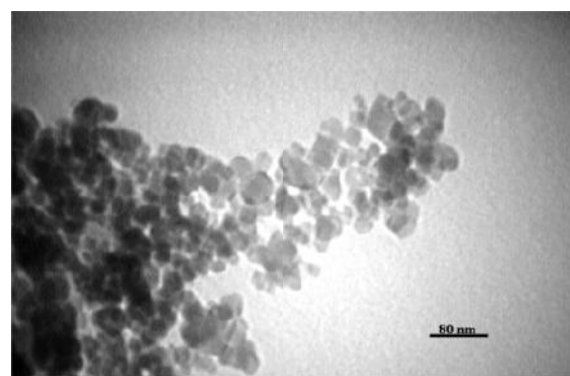


Figure 3. TEM image of the Fe_3O_4 nanoparticle

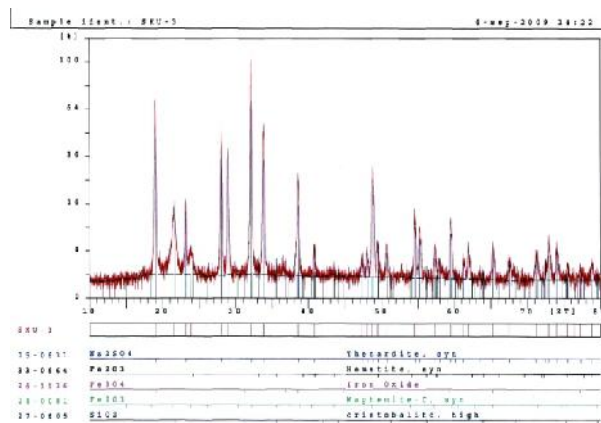


Figure 4: XRD pattern of the Fe₃O₄ nanoparticle

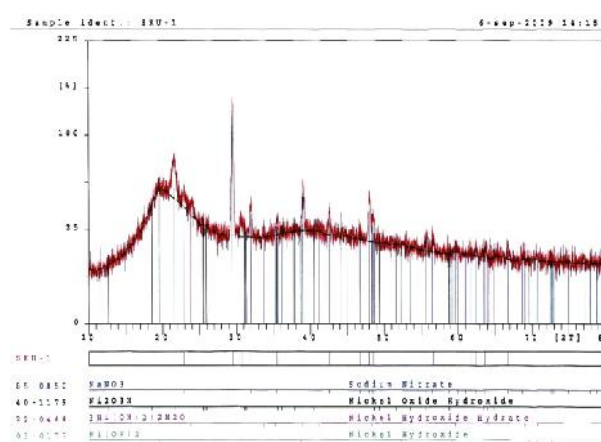
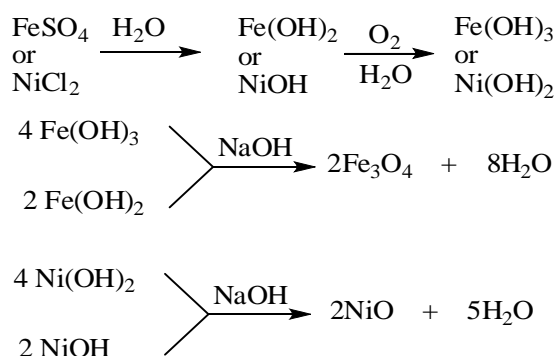


Figure 5: XRD pattern of the NiO nanoparticle

Discussion

In the present study, Fe₃O₄ (or NiO) formation in this process may proceed through two steps. Firstly, Fe(OH)₂ (or NiOH) is formed in aqueous solution when FeSO₄·7H₂O (or NiCl₂·6H₂O) is dissolved in H₂O, and a fraction of Fe(OH)₂ (or NiOH) is oxidized to Fe(OH)₃ (or Ni(OH)₂) by the oxygen in the environment. Finally, Fe(OH)₂ (or NiOH) and Fe(OH)₃ (or Ni(OH)₂) deposit with each other in the basic conditions and then Fe₃O₄ (or NiO) nanostructures are obtained. This process is presented by a chemical equation as follows:



Conclusion

In this research we have described the use of an inorganic phase in water-in-oil (w/o) microemulsion has received considerable attention for preparing nanomagnetic iron and nickel oxides via a sol-gel reaction in reverse micelles from FeSO₄ and NiCl₂ source at room-temperature. The nanoparticles with homogeneous size, shape distribution agglomeration of particles, with diameters ranging from 40 to 60 nm have been characterized by SEM, TEM and the XRD. In addition, the easily controllable conditions with using low cost iron and nickel source is merit to be considered for scaling up by industrial researchers. In the present study synthesis of nanoparticles can be easily implemented because it is simple and environmentally friendly.

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