Original Article
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Green synthesis and characterization of $NiFe_2O_4$ nanostructure via hydrothermal method

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Abstract
In this paper, nickel ferrite nanostructures were fabricated via a simple and green hydrothermal method using lemon juice. For the synthesis of $NiFe_2O_4$ nanoparticles, the materials were placed in the autoclave reactor for 2 and 12 hours. The morphology of particles was studied by scanning electron microscope (SEM). Although the $NiFe_2O_4$ nanoparticles after 2 h heating are agglomerated increasing the heating time to 12 h leads to the formation of sphere shape nanosize particles. The crystal structure of nickel ferrite nanoparticles was investigated via an X-ray diffraction pattern and the crystallite size of particles was calculated using the Debye-Scherrer formula. The magnetic hysteresis loop of the synthesized ferrite was studied by a vibration sample magnetometer (VSM). The magnetic parameters of remanence Mr, saturation magnetization Ms, and coercivity Hc, increase as the synthesis time increases. The optical properties of $NiFe_2O_4$ were investigated by ultraviolet-visible (UV-vis) spectra. The results show that the particle band gaps decrease with increasing the synthesis time.

Keywords: $NiFe_2O_4$, Hydrothermal, Lemon juice

1. Introduction

The ferrite nanoparticles have unique structural, physical and magnetic properties and have many applications in microwave and recording devices, sensors, magnetic refrigerators, photoelectric devices, magnetic pigments and high-frequency devices [1-4].

The spinel ferrites are a kind of soft magnetic material. The spinel ferrites have the AFe2O4 formula, that A is a divalent ion like Ni, Co, Mn, Zn and etc. These ferrites have low saturation magnetization and coercivity and high expansion coefficients [5, 6]. Nickel ferrite is a famous soft spinel ferrite with low coercivity. The $NiFe_2O_4$ has an inverse spinel structure. In this structure, the iron ions are located in octahedral and tetrahedral sites and nickel ions are replaced only in octahedral sites. The magnetic properties of nanocrystalline $NiFe_2O_4$ are different from bulk nickel ferrite and depend on the size, shape, and purity of nanoparticles, there for these nanoparticles are interested in many industrial and scientific applications [7-9]. There are various methods for synthesizing $NiFe_2O_4$ nanocrystalline such as sol–gel, microemulsion, microwave, sonochemical, and hydrothermal [10-14]. between these methods, the hydrothermal process is very interesting because of its cost-effectiveness and control of the morphologies and sizes of particles by control of hydrothermal parameters like time temperature, and concentration of materials [15]. In the hydrothermal method, the nanomaterials can be synthesized in a large temperature range from the evaporation temperature of water (about 373 K) to Temperatures above the evaporation rate of water (about 623 K). The increased temperature cause increases the pressure in the autoclave reactor [16].

The hydrothermal method can fabricate nanostructures that are not stable in high temperatures. The nanomaterials that have been synthesized via the hydrothermal method can be well controlled in liquid or other phase chemical phase reactions [17].

In this paper, the $NiFe_2O_4$ nanostructures synthesized via the hydrothermal method and the effect of autoclave heating time on structural and magnetic properties were investigated.

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2. Experimental

2.1. Material

$Ni(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$ and NaOH were prepared from Merck Co. All materials had purity above 99%. The deionized water and lemon juice have been used as a solvent as a capping agent respectively.

2.2. Preparation of the $NiFe_2O_4$ nanoparticles

$Ni(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ were dissolved in deionized water with a molar ratio of 1:2. Then lemon juice then was added to the solution with a volume ratio of 1:5. NaOH (1 M) then was added to the solution drop by drop until the pH is maintained at 11. The mixture is stirred for 30 min, and then it was spilled at a stainless-steel autoclave with 500 ml capacity at 180°C for 2 and 12 h. The resulting black material was centrifuged and washed with distilled water. Finally, it was placed in an oven with a temperature of 80°C for 24 h to dry. The schematic diagram of the experimental setup was shown in Fig. 1.

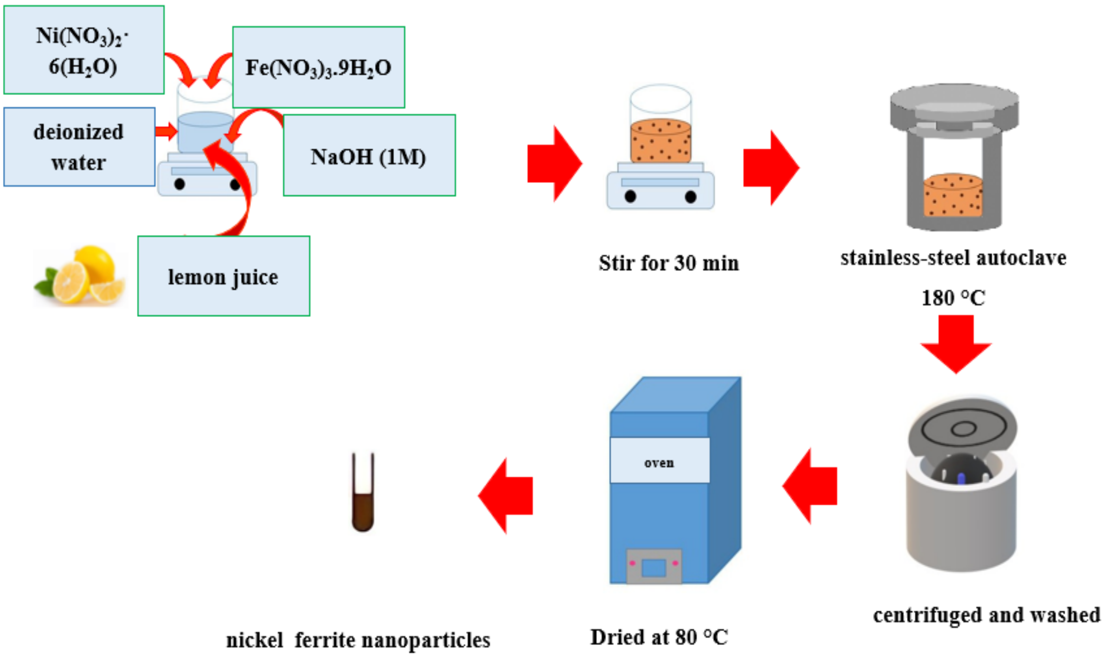


Fig. 1. Schematic of nickel ferrite preparation via hydrothermal method.

2.3. Characterization

The hysteresis curve of particles was investigated via a VSM device (Model force-MDKF made by a Magnetic Danesh Pajoh Co., Measured in the central lab of Arak University) at room temperature in a magnetic field varying between ±10000 Oe. XRD patterns curve were studied using a Philips X-ray diffractometer (with the wavelength of 1.54 Å from CuKα radiation). SEM images were obtained using an LEO device model 1455VP.

3. Results and discussion

3.1. XRD analysis

Fig. 2 (a). shows the XRD pattern of the $NiFe_2O_4$ particles fabricated by hydrothermal method at180°C for 2 h. The XRD peaks examined the cubic crystal system which is completely compatible with the standard card JCPDS No.: 00-003-0875. The X-ray diffraction pattern of the nickel ferrite particles that was synthesized by hydrothermal method at180°C for 12 h is presented in Fig. 2 (b). The average particle crystalline size is calculated by the Scherrer equation, (Eq. 1), that β is the width of the observed diffraction peak at its half maximum intensity, λ is the x-ray wavelength, 0.154 nm and θ is the diffraction position [18]. The average crystalline size for $NiFe_2O_4$ particles synthesis for 2 and 12 h heating time were found to be about 29 and 58 nm respectively.

$$D = 0.9\lambda/\beta\cos\theta \quad (1)$$

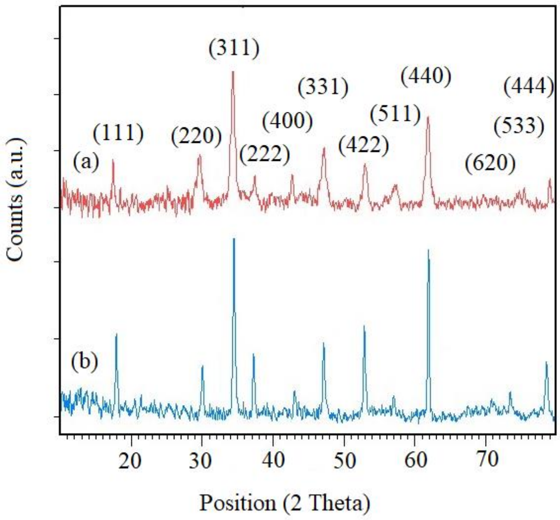
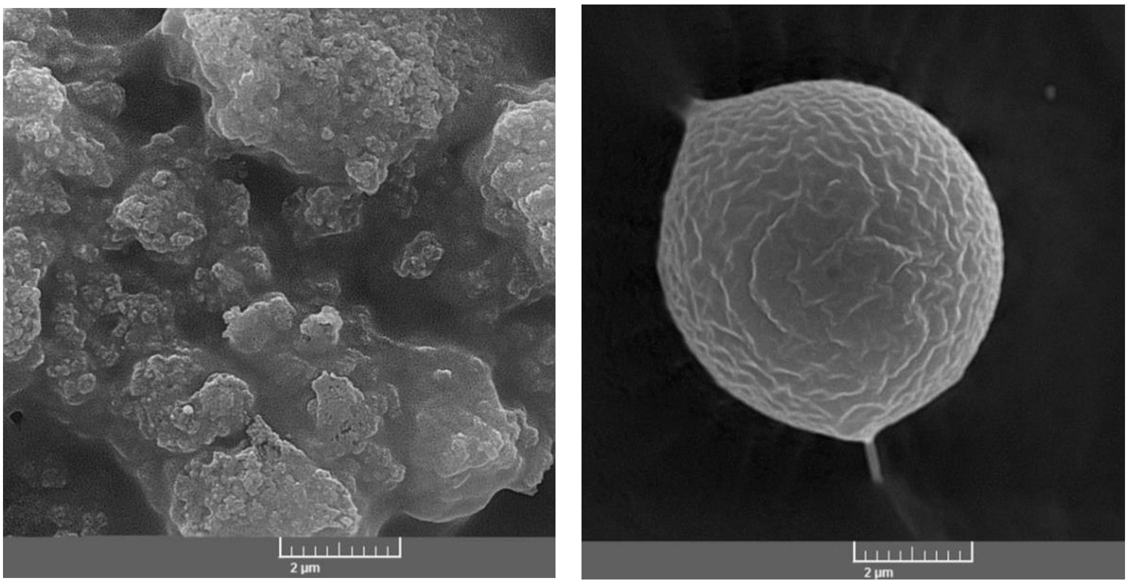


Fig. 2. XRD pattern of $NiFe_2O_4$ nanoparticles synthesized by hydrothermal method at180°C for (a) 2 and (b) 12 h.

3.2. SEM analysis

SEM images of $NiFe_2O_4$ nanoparticles fabricated at 180 °C for 2 h are illustrated in Fig. 3 (a). SEM image approves the formation of agglomerated nanostructures with a size of less than 100 nm. The morphologies of $NiFe_2O_4$ nanoparticles synthesized at 180 °C for 12 h are shown in Fig. 3 (b). The SEM image shows a giant sphere with raised grooves in a range of less than 100nm. The SEM images show that increasing the heating time leads to some orders in the formation of nanostructures produced by the hydrothermal method. The reason for the regularization of the structure during the longer synthesis time is to have more opportunity for crystal growth at high temperatures.



(a) **(b)**

Fig. 3. SEM images of $NiFe_2O_4$ nanoparticles synthesized by hydrothermal method at 180°C for (a) 2 h and (b) 12 h.

3.3. VSM analysis

Room temperature magnetic property of $NiFe_2O_4$ nanoparticles was studied using a VSM instrument and the obtained curves are shown in Fig. 5. The hysteresis loop of $NiFe_2O_4$ exhibits ferromagnetic behavior with coercivity, remanence magnetization, and saturation magnetization that are summarized in table 1. The values indicate that with the increase in the synthesis time in the hydrothermal method, the magnetic properties become stronger and the Mr, Ms, and Hc increase as well.

Tab. 1. M_r, M_s and H_c for $NiFe_2O_4$ nanoparticles

Synthesis time (h)	Remanence magnetization (emu/g)	Saturation magnetization (emu/g)	Coercivity (Oe)
2	1.81	8.95	250
12	3.25	11.79	475

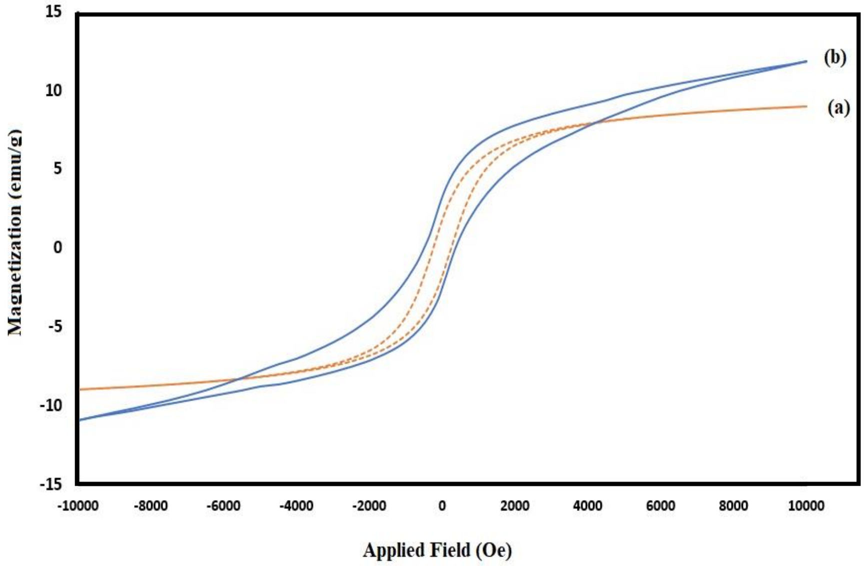


Fig. 5. Hysteresis loop of $NiFe_2O_4$ nanoparticles synthesized by hydrothermal method at 180°C for (a) 2 and (b) 12 h.

3.4. UV-Vis analysis

The UV-vis absorbance spectra of $NiFe_2O_4$ nanoparticles fabricated by hydrothermal method at 180 °C for 2 and 12 h are illustrated in Fig. 6 (a) and (b) respectively. The graphs show that for the synthesis time of 2 h the band gap is 4.96 eV and for 12 h the band gap is 4.35 eV. The reason for decreasing the energy gap could be due to an increase in the grain size with increasing the synthesis time.

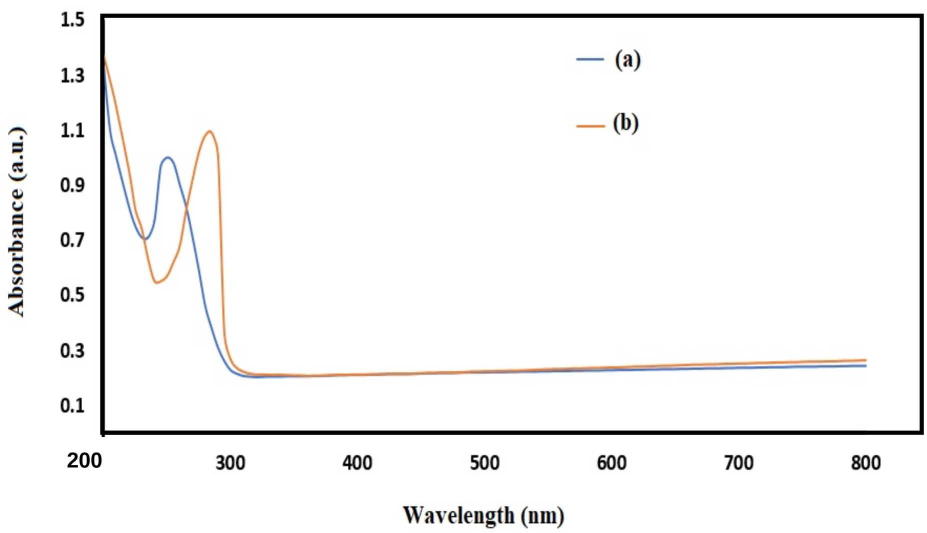


Fig. 6. Absorbance spectra of $NiFe_2O_4$ nanoparticles synthesized by hydrothermal method at180°C for (a) 2 and (b) 12 h.

4. Conclusions

The nickel ferrite nanostructures were synthesized using a simple hydrothermal method at 180 °C for 2 and 12 h. The average crystalline size for $NiFe_2O_4$ particles increases by increasing the heating time. The XRD peaks are completely compatible with the standard card JCPDS for nickel ferrite. The morphologies of $NiFe_2O_4$ for 2 h heating time approve the formation of agglomerated nanostructures while by increasing the synthesis time to 12 h sphere shape particles with nanosize raised grooves are obtained. The Mr, Ms, and Hc values increase with the increase in the synthesis time. The band gap of particles decreases with an increase in the synthesis time.

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