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Investigating the Effect of Ultrasound Intensity on the Magnetic Properties of Magnetite Nanostructures Synthesized by Sonochemical Method

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ABSTRACT

In this article, the synthesis of magnetite nanostructures was successfully carried out by the sonochemical process. In this method, stoichiometric amount of iron chlorides ($FeCl_3.6H_2O$ and $FeCl_2.4H_2O$), ammonia (NH_3) and polyvinylpyrolidone (PVP) were used to synthesize pure Fe_3O_4 nanoparticles. The effect of initial sonication power of the ultrasonic device on the size and morphology of the final products as one of the effective parameters was investigated. For this, the initial power of the sonicator was evaluated at 90, 70, 50 and 30 W at 40°C. Characterization of Fe_3O_4 nanoparticles was done by transmission electron microscope (TEM) and X-ray powder diffraction (XRD) and its magnetic properties were investigated by vibrating sample magnetometer (VSM). Investigation of the XRD pattern after annealing showed that pure Fe_3O_4 phase was successfully formed during the sonochemical process. TEM images determined the size of Fe_3O_4 nanoparticles to be 10-50 nm. The results showed that increasing the initial power of the system reduced the particle size and improved the magnetic properties of nanoparticles.

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1. INTRODUCTION

The materials properties depend on the kind and situation of atoms in the structure. When the materials dimension approaches molecular and atomic size, the ratio of area to volume increases considerably. Therefore, the behavior of nanomaterials is substantially different from bulk microstructures [1-3].

Iron oxide nanoparticles with various structures, known as very important nanomaterials, have been used broadly in many fields such as electrical topics, magnetism, medicine, chemical, dying and the food industry. Fe_3O_4 nanostructures have various applications in fabricating sensors, shape memory alloys, rechargeable lithium batteries and drug delivery because of their unique chemical and magnetic characteristics [4, 5]. These nanomaterials attracted to magnets have been studied as drug delivery factors. Fe_3O_4 nanoparticles have attracted much attention from researchers due to their superparamagnetic properties, and containing both Fe(II) and Fe(III) ions, which makes it superior to other types of iron oxides [6].

Fe_3O_4 nanoparticles are also used in infectious diseases, encapsulating medicine, active drug delivery, applied sensors, diagnostic cells, hyperthermia, making genes and imaging of the body and determining the diseases via increasing the image contrast in MRI as well. This kind of particle ought to be enough small in order to prevent them to be deposited and also remaining invisible against the phagocytosis systems [3, 7]. Despite the weak and strong points of iron oxides in practical applications, superparamagnetic iron oxides and super paramagnetic iron nanoparticles are the only magnetic nanoparticles used in medical applications [8]. Materials magnetic properties result from the electrons magnetic moments. Each electron has a magnetic moment in the atom that

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comes from two sources: one is related to the orbital motion around the nucleus (electron orbit magnetic moment), and another is associated with the electron spinning around its axis (electron spin magnetic moment) [9]. Furthermore, each electron can be a small permanent magnet using its orbit and spin magnetic moments [10]. The relation between the properties and the grain size is a very important issue in magnetic materials. In the case of soft magnetic materials, the area under the hysteresis curve significantly decreases with reducing the size of grains even if it can be disappeared completely [11, 12].

So far, various synthesis methods have been mentioned for the synthesis of magnetite nanoparticles, including the sol-gel process, chemical vapor deposition (CVD), hydrothermal technique, pulsed laser evaporation, electron irradiation, and reactive sputtering [12-15]. However, chemical processes are often used because it is an easy, repeatable and cost-effective methods, and among them, chemical bath deposition (CBD) and sonochemical processes are more useful [16-18]. In previous works, Fe_3O_4 nanoparticles have been synthesized by the sonochemical method. The results have shown that ultrasonic radiation causes cavitation in an aqueous medium, which leads to the formation, growth, and collapse of microbubbles [19, 20]. Therefore, the quality and properties of the synthesized product will be affected. In this work, the effect of ultrasound intensity on the structural and Fe_3O_4 properties of magnetite nanoparticles synthesized by the sonochemical method will be investigated.

2. MATERIALS AND EXPERIMENTS

2. 1. Materials In this work iron chlorides ($FeCl_3.6H_2O$, $FeCl_2.4H_2O$), ammonia (NH_3) and polyvinylpyrrolidone (PVP) were purchased from Merck.

2. 2. Synthesis The synthesis of magnetite nanoparticles was done according to the previous method [18]. In this method, $FeCl_3.6H_2O$ (0.4 M, 50 ml) and $FeCl_2.4H_2O$ (0.2 M, 50 ml) are used as starting precursors and PVP as a surfactant, and ammonia (0.8 M, 50 ml) is also used. NH_3 was added dropwise to the resulting aqueous solution. Process sonication was provided using the Misonix sonicator model S-4000 sonicator through direct ultrasonic irradiation with an initial power of 10, 30, 50 and 70 W for about 30 min and a temperature of 40 °C. Then the solution was exposed to ultrasound for 60 min to complete the sonochemical process. The influence of ultrasonic power on the structure and morphologic properties of nanoparticles has been studied and samples I, II, III, and IV are prepared under different conditions listed in Table 1.

After completing the procedure and appearing precipitations, the obtained solution is put in the centrifuge system at 1000 rpm for 15 min to separate precipitations containing nanoparticles. After collection, the final precipitated particles were filtered and washed with methanol and distilled water to remove by-products. Then all the samples were air-dried at room temperature and calcined at 500 °C for 60 min to obtain crystalline iron oxide nanoparticles.

TABLE 1. The process conditions of ultrasonic device for synthesizing magnetite nanoparticles

Sample Code	Initial Power (W)	Ultrasonic Waves Intensity (W/cm^2)	Imported Energy into the Solution (kJ)
I	30	52	123
II	50	65	150
III	70	78	185
IV	90	94	202

2. 3. Characterizations Crystal phases of Fe_3O_4 nanoparticles were characterized by X-ray diffraction (Siemens D-5000, with Cu Kα radiation). The morphology of the synthesized samples was studied by TEM analysis (ZEISS, Germany). Their magnetic properties were measured with a vibrating sample magnetometer system (AGSM model, ± 0.001 emu/g).

3. RESULTS AND DISCUSSIONS

The primary product without annealing has a completely amorphous structure [18, 21]. This is due to the collapse of high-temperature bubbles in some parts of the solution where the bubbles are present, so as the temperature decreases rapidly, the particles do not have enough time to crystallize. Under these conditions, the reactants carry out intense interactions in a very short period of time. Cavitation events lead to increased heating and cooling rates to more than 10^{10} Ks^{-1} [22, 23]. Hence, after TGA/DTA analysis in previous work and determining the crystallinity temperature, the samples were heat-treated at 500 °C for about 1 h to gain a calcined powder and also to get the required peaks for phase and structure analysis [18]. It can be seen in Figure 1 which is related to the diffraction of Fe_3O_4 nanoparticles, all peaks are associated with Fe_3O_4 nanoparticles and there are not any peaks associated with impurities. The sharp peaks with high intensity show the high crystallinity of the prepared nanopowder via sonochemical method.

Considering the pattern, it can be found that there is a direct relation between ultrasonic intensity and flatness of peaks. As much as the initial power is raised, flatness of peaks is also increased, although the crystallite size will be decreased, in sequence. Using the XRD patterns and Scherer equation, the size of the crystals was determined [24]:

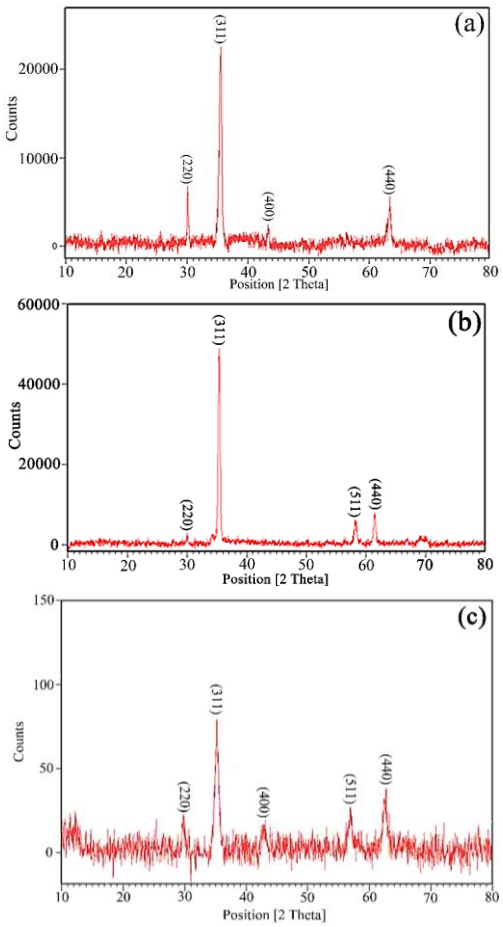

Figure 1. XRD patterns of the samples (a) I, (b) II, and (c) IV

$$D = \frac{k.\lambda}{\beta.\cos\theta} \quad (1)$$

where k is constant (0.89 for spherical particles), D is the crystallite size (nm), λ is the wavelength of the X-ray beam (λ= 0.15406 nm for Cu Kα radiation), β is the full width at half maximum for the diffraction peak under consideration (in radians), according to measurements, the mean size of crystals is about 8 nm, 14 nm, and 17 nm for initial powers of 90, 50 and 30 W, respectively. Ultrasonic waves disperse the suspension solution into smaller droplets. In this way, it reduces the kinetics of particle growth and the amount of aggregation and stops crystal growth. This leads to reduced crystallinity, cluster decomposition [25]. The ultrasonic process is more effective than the magnetic field in the particle growth step, and the bubbles formed in this process break more particles into smaller particles [23].

Figure 2 shows TEM images of Fe_3O_4 nanostructures prepared via sonochemical process with different powers. Fe_3O_4 nanoparticles have spherical morphology with smooth geometry and high degree of crystallinity.

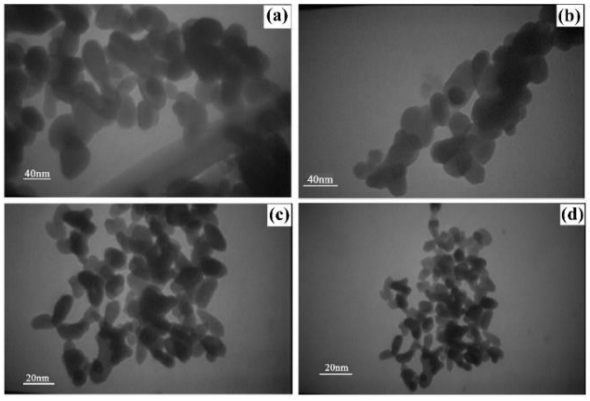

Figure 2. TEM images of Fe_3O_4 nanoparticles at initial powers: (a) I, (b) II, (c) III, (d) IV

The gained size of nanoparticles is shown in Table 2. Cavitation promotes the local reaction, heating, and severe stirring through the heat transfer of gas bubbles. Therefore, by accelerating the nucleation process, the concentration of seeds increases. This phenomenon leads to the relaxation and interface defects and the formation of nanoparticles [22].

Since the temperature and solution concentration are constant for all samples in this research, these factors have not influenced the results and the only effective factor is ultrasonic wave intensity. By increasing the initial power, the bubbles are formed with more energy. Furthermore, the sonochemical influences of this kind of bubbles will increase. These bubbles have more energy so they have more sonochemical influences on the solution [26-28].

According to Table 1, by increasing the initial power from 30 to 50 W at constant temperature of 40 °C, the intensity of the ultrasonic waves has increased from 52 W/cm^2 to 94 W/cm^2. This increase is due to the expansion of energetic bubbles or the increase in the solution viscosity due to precipitation formation.

The VSM analysis is used in order to determining the effect of the ultrasonic waves intensity on the magnetic properties of the prepared nanostructures via sonochemical technique. Figure 3 shows the hysteresis curves of the prepared nanostructures, and their results are listed in Table 3.

TABLE 2. The average size of obtained Fe_3O_4 nanostructure measured from TEM images

Sample Code	The size of particles (nm)
I	50
II	35
III	20
IV	10

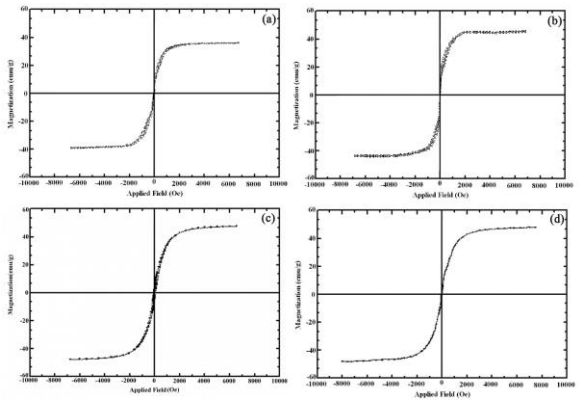

Figure 3. VSM spectra of the synthesized samples: (a) I, (b) II, (c) III, (d) IV

TABLE 3. Magnetic properties of the samples synthesized via sonochemical processing with different intensity of ultrasonic waves

Sample Code	Initial power (W)	M (emu/g)	H_C (Oe)
I	30	38	1100
II	50	43	1000
III	70	47	800
IV	90	50	-

It can be seen that by increasing the power and the intensity of ultrasonic waves, the magnetism of particles has decreased. Reduction in the particle size results in raising coercivity to a maximum amount (IV), then it will be decreased until it will be closed to zero. Yadav et al. [29] showed that increasing of the ultrasonic power input in the synthesis of $MnFe_2O_4$ increases the saturation magnetization and negligible surface spin canting.

The main reason for this situation is that the magnetization of the multi-domain nanoparticles can be changed with the motion of the domain walls. In this case, nanoparticles with small sizes consist of one domain, but larger particles that are composed of several domains minimize the static magnetization energy. The approximate range for the critical particle size above which the magnetic nanoparticle is not single-domain is from a few to several tens of nanometers, which is higher than the typical domain wall dimension in the magnetic material. The nature of the domain structure has a strong influence on the hysteresis behaviour of magnetic nanoparticles. By decreasing the ultrasonic frequency, the nucleation rate accelerates due to an increase in the magnitude of the shock wave caused by the cavitation collapse. Therefore, magnetization improves as the crystallite size decreases [30].

Considering the XRD and TEM results of obtained samples have been prepared via the sonochemical process, it is clear that decreasing the particle and crystallite sizes tends to reduce the coercivity to zero. Therefore, by controlling the particle size in the nano dimension, it is possible to change the coercivity easily. After removing the magnetic field, the magnetic properties are disappeared approximately in this kind of nanostructure. If permanent magnets will be necessary, nanopowders should keep their magnetite properties even in absence of a magnetic field. Consequently, the hysteresis loop should be large, which means that H_c and M_r should be high, but the hysteresis loop of the nanostructures obtained via the sonochemical process is small. It is an important point that declares a good relationship between the properties and the grain size of materials. In soft magnetic materials decreasing the grain size tends to reduce the area under the hysteresis graph significantly, even if it may disappear completely such as in sample IV. Fuentes-García et al. [31] synthesized magnetite nanoparticles by a one-step sonochemical method. The results showed that the obtained nanomaterials with improved properties have a good potential to be used as magnetic hyperthermia agents.

4. CONCLUSIONS

In this work, the sonochemical process was used as a new and advanced technique with direct ultrasonic waves irradiating on the solution for the synthesis of iron oxide (Fe_3O_4) nanostructures. Then, the effect of the intensity of ultrasonic waves on the properties of Fe_3O_4 nanoparticles was investigated. The obtained nanoparticles have an amorphous structure resulting from the collapse of cavitation bubbles as enormous cooling rate. Because it prevents their crystallization during quenching and also the quick decrease of temperature around the nanoparticles. Therefore, to obtain crystallite nanoparticles, they must be calcined. The significant results of this work relate to the size, morphology, structure, purity and magnetic properties of the prepared materials. The size of crystallites is about 8 nm to 17 nm and the particle size considering the average size of particles is 10-50 nm which put them in a super small iron oxide nanoparticles group with a diameter of less than 50 nm. It can be seen that increasing the initial power of the system reduces the grain size with which the magnetic properties of particles have increased. Synthesized magnetite nanoparticles have a spherical morphology with smooth geometry and a degree of crystallinity, which are very useful and practical in various fields.

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Persian Abstract

چکیده

در این مقاله، سنتز نانوذرات مگنتیت با فرآیند سونوشیمیایی با موفقیت انجام شد. در این روش از مقدار استوکیومتری کلریدهای آهن ($FeCl_3.6H_2O$) و ($FeCl_2.4H_2O$)، آمونیاک (NH_3) و پلی وینیل پیرولیدون (PVP) برای سنتز نانوذرات خالص Fe_3O_4 استفاده شد. تاثیر توان امواج آلتراسونیک اولیه بر اندازه و مورفولوژی محصولات نهایی به عنوان یکی از پارامترهای موثر مورد بررسی قرار گرفت. برای این کار، توان اولیه دستگاه سونیکاتور در ۹۰، ۷۰، ۵۰ و ۳۰ وات در دمای ٤٠ درجه سانتیگراد ارزیابی شد. شناسایی نانوذرات Fe_3O_4 با استفاده از میکروسکوپ الکترونی عبوری (TEM) و پراش پرتو ایکس (XRD) و خواص مغناطیسی آن توسط مغناطیس سنج نمونه ارتعاشی (VSM) بررسی شد. بررسی الگوی XRD پس از کلسینه کردن نشان داد که فاز Fe_3O_4 خالص با موفقیت در فرآیند سونوشیمیایی تشکیل شد. تصاویر TEM اندازه نانوذرات Fe_3O_4 را ۱۰-۵۰ نانومتر تعیین کردند. نتایج نشان داد که افزایش توان اولیه سیستم باعث کاهش اندازه ذرات و بهبود خواص مغناطیسی نانوذرات می‌شود.