



Synthesis and Characterization of Magnetic Fe₃O₄ Nanoparticles by Laser Ablation Technique

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ABSTRACT: In recent years, significant attention has been devoted to the development of physical, chemical, and biological methods for synthesizing nanomaterials. While chemical methods typically offer faster synthesis rates compared to physical and biological approaches, they are often associated with environmental hazards and lower product purity. Among the physical techniques, laser ablation has emerged as a promising and clean method, wherein high-energy laser pulses are employed to remove material from a solid target. In this work, magnetic iron oxide (Fe₃O₄) nanoparticles were synthesized via laser ablation in an aqueous medium. A fiber laser operating at a wavelength of 1064 nm was used to irradiate a high-purity (99.99%) iron target. The structural and magnetic characteristics of the resulting nanoparticles were characterized using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), and vibrating sample magnetometry (VSM). Additionally, Fourier-transform infrared spectroscopy (FTIR) was employed to identify functional groups present in the sample. The XRD pattern confirmed the formation of pure Fe₃O₄ nanoparticles, showing full agreement with standard reference data. FESEM analysis revealed spherical nanoparticles with diameters ranging from 23 to 30 nm. Magnetic measurements indicated a saturation magnetization value of 28.32 emu/g, confirming the magnetic nature of the synthesized nanoparticles.

KEYWORDS: Fe₃O₄, Laser ablation technique, Magnetic nanoparticles, Nanomaterials Zharacterization, Physical synthesis methods.

INTRODUCTION

Nanoparticles are materials with dimensions ranging from 1 to 100 nm and may be composed of carbon-based substances, metals, metal oxides, or natural materials. Compared to their bulk counterparts, nanoparticles exhibit unique physical, chemical, and biological properties. Their exceptionally high surface-area-to-volume ratio, enhanced reactivity, chemical stability, and increased mechanical strength are among the remarkable characteristics of nanoparticles [1], [2]. These distinctive properties have led to the wide application of nanoparticles in various fields— for example, in drug delivery and transport systems [3], in the removal of water pollutants [4], and in the development of catalysts for energy production and storage [5], among others. Nanoparticles also differ in their size, shape, and dimensionality. For instance, graphene is considered a two- dimensional nanomaterial, while carbon nanotubes and gold nanoparticles are classified as three- dimensional nanomaterials [2].

Recently, the use of magnetic nanoparticles in various industrial and medical applications has gained significant attention due to their unique physical, chemical, optical, and electrical properties. For example, magnetic nanoparticles exhibit remarkable catalytic performance in the removal of pollutants and are widely used in water purification processes. Among magnetic nanomaterials, iron oxide nanoparticles are particularly important. Although iron oxide nanoparticles can exist in several crystalline phases, the most well-known magnetic phases are magnetite (Fe₃O₄) and hematite (Fe₂O₃). These two common phases exhibit different physical and chemical properties, which arise from variations in the oxidation states of iron. Magnetite (Fe₃O₄), a black ferromagnetic iron oxide composed of Fe(II) and Fe(III), is the most widely used and studied form of iron oxide. Due to the presence of Fe²⁺ ions, magnetite can act as an electron donor [6]. The magnetic properties of magnetic nanoparticles are characterized based on their response to an external magnetic field [7]. In general, magnetic materials are classified based on their behavior in an external magnetic field as diamagnetic, paramagnetic, ferromagnetic, antiferromagnetic, or ferrimagnetic [8].

In a paramagnetic material, the partial alignment of existing magnetic dipole moments is the primary source of magnetization. In the absence of an external magnetic field, these dipoles are randomly oriented. However, when an external magnetic field is applied,

the magnetic dipole moments tend to align with the direction of the field, thereby enhancing the overall magnetic effect. In contrast, when an external magnetic field is applied to a diamagnetic material, the atomic orbitals are altered due to changes in the motion of the electrons. As a result, an induced magnetic field is spontaneously generated that opposes the change in the external magnetic field, in accordance with Lenz's law. Therefore, the diamagnetic response originates from an induced current that produces a magnetic field opposite to the applied one. In antiferromagnetic materials, neighboring magnetic moment vectors have equal magnitudes but are oriented in opposite directions, thereby canceling each other out. These materials typically exist at low temperatures; however, at higher temperatures, they may exhibit ferromagnetic or diamagnetic behavior. In ferrimagnetic materials, the magnetic moments are also oriented in opposite directions but differ in magnitude. As a result, these materials behave similarly to ferromagnetic substances and can retain their magnetization even after the removal of an external magnetic field. Ferrimagnetic materials become paramagnetic above a certain temperature, known as the Curie temperature. Ferromagnetic materials, such as magnetite, possess permanent magnetic moments even in the absence of an external magnetic field and exhibit strong magnetization. In these materials, the magnetic dipole moments are aligned parallel to each other. Magnetic iron oxide nanoparticles can be synthesized using various physical (top-down and bottom-up), chemical (bottom-up), and biological methods [9]. Among these, laser ablation is considered a clean, rapid, simple, and cost-effective physical technique [10].

In the liquid-phase laser ablation method, a focused laser beam is directed onto the surface of a target submerged in a liquid, leading to the atomization and ionization of the target as well as the decomposition of the liquid. As a result, plasma is generated on the surface of the target, and its expansion is confined by the surrounding liquid. Under high temperature and pressure conditions, chemical reactions occur between the species present in the plasma and the ionized species in the liquid environment. Subsequently, the plasma extinguishes, and a cavitation bubble forms, which contains the laser ablation products. With increasing temperature and pressure, the cavitation bubble collapses, dispersing the ablation products into the liquid medium[11]. Figure 1 schematically illustrates the laser ablation process in a liquid environment.

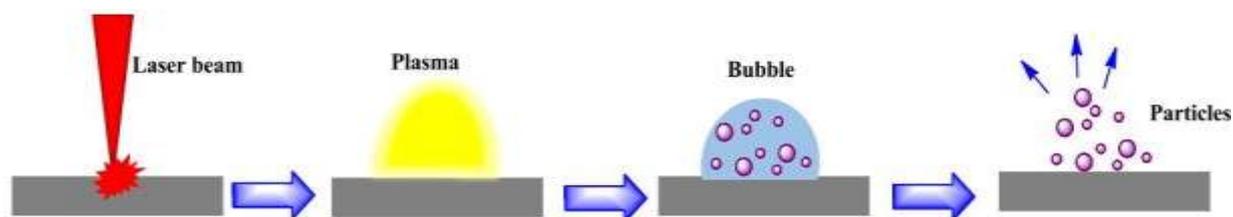


Figure 1. Schematic diagram of the laser ablation process in a liquid environment.

In this study, iron oxide nanoparticles were synthesized from a pure iron target using the liquid-phase laser ablation method. The structural and magnetic properties of the resulting nanoparticles were characterized using FESEM, FTIR, XRD, EDS, and VSM analyses.

2. EXPERIMENTAL PROCEDURE

First, the surface of the iron target was polished using sandpaper and then cleaned in acetone for 15 minutes using an ultrasonic bath. After rinsing with deionized water, the target was again cleaned for 15 minutes in the ultrasonic bath while submerged in deionized water. Subsequently, while remaining submerged in deionized water, the target was irradiated with a laser beam. In the laser ablation process, a fiber laser (RFL-P30Q) with a wavelength of 1064 nm, a maximum power of 30 W, and a frequency of 20 kHz was used. The laser beam was focused onto the target surface, measuring $18 \times 25 \text{ cm}^2$, through a $40 \mu\text{m}$ quartz lens. With increasing irradiation time, the color of the deionized water gradually turned dark brown, indicating the formation of nanoparticles. During the experiment, the laser irradiation was paused every 10 minutes to prevent an increase in the ambient temperature. Once the deionized water in the irradiation chamber reached saturation, the colloidal nanoparticles were transferred to a separate beaker to prevent a decrease in laser efficiency and nanoparticle agglomeration. Finally, the nanoparticles were separated from the deionized water using a strong magnet and dried at room temperature.

3. DISCUSSION AND ANALYSIS

The functional groups present in the synthesized sample were investigated using FTIR analysis. Figure 2 displays the resulting spectrum in the range of 4000–400 cm^{-1} . The absorption observed in the region of 3421–3000 cm^{-1} is attributed to the symmetric and asymmetric stretching vibrations of O–H. The peaks appearing in the wavenumber range of 1630–1341 cm^{-1} correspond to the H–O–H bending vibrations. Additionally, the absorption at 591 cm^{-1} is assigned to Fe–O vibrations, confirming the magnetic nature of the sample [12].

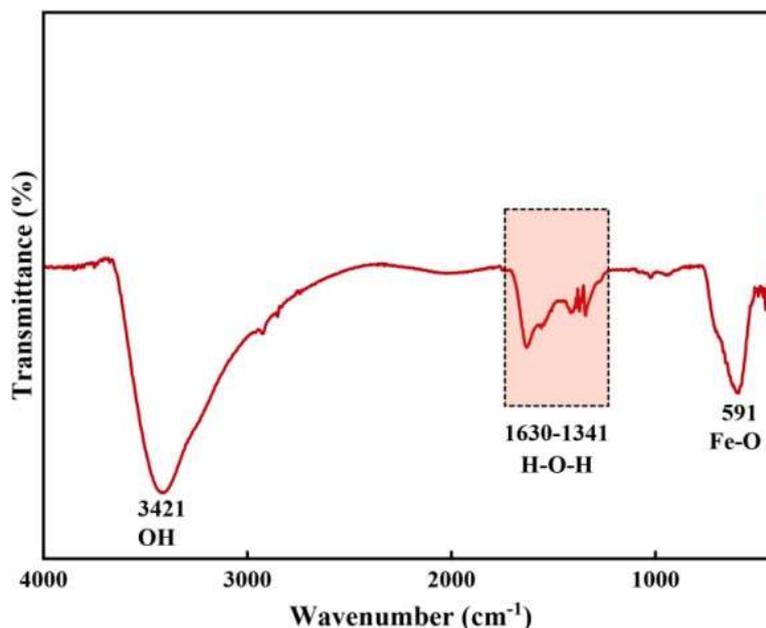


Figure 2. FTIR spectrum of the synthesized nanoparticles.

For the purpose of analyzing and investigating the structure of the synthesized nanomaterial, XRD analysis was employed. According to the diffraction pattern shown in the figure, three peaks appearing at angles 35.6° and 63.6° correspond to the diffraction from the (311) and (440) planes, respectively (JCPDS 75-0033). Additionally, the peak observed at 44.8° to the presence of elemental iron (Fe)

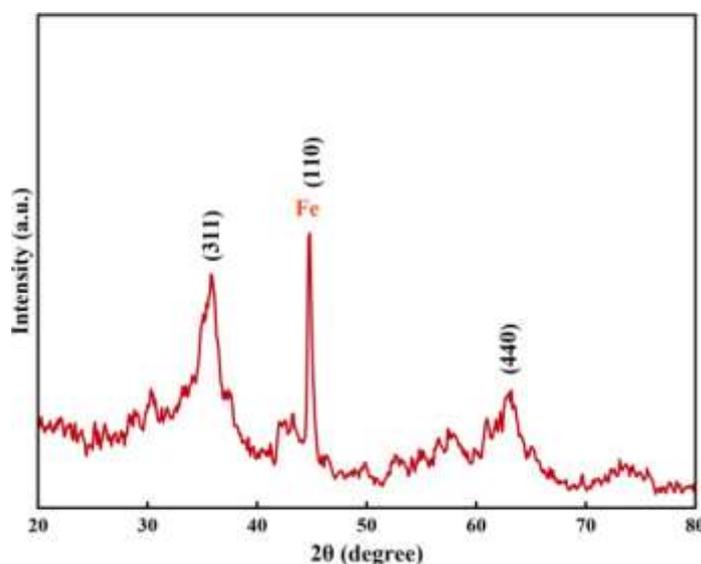


Figure 3 – XRD pattern of the synthesized sample.

SEM was used to determine the structure of the synthesized Nano rods. As shown in Figure 4, spherical iron oxide Nano rods were successfully produced by the pulsed laser ablation method in a liquid medium. According to Figure 4b, the synthesized spherical Nano rods have diameters ranging from 23 to 30 nm.

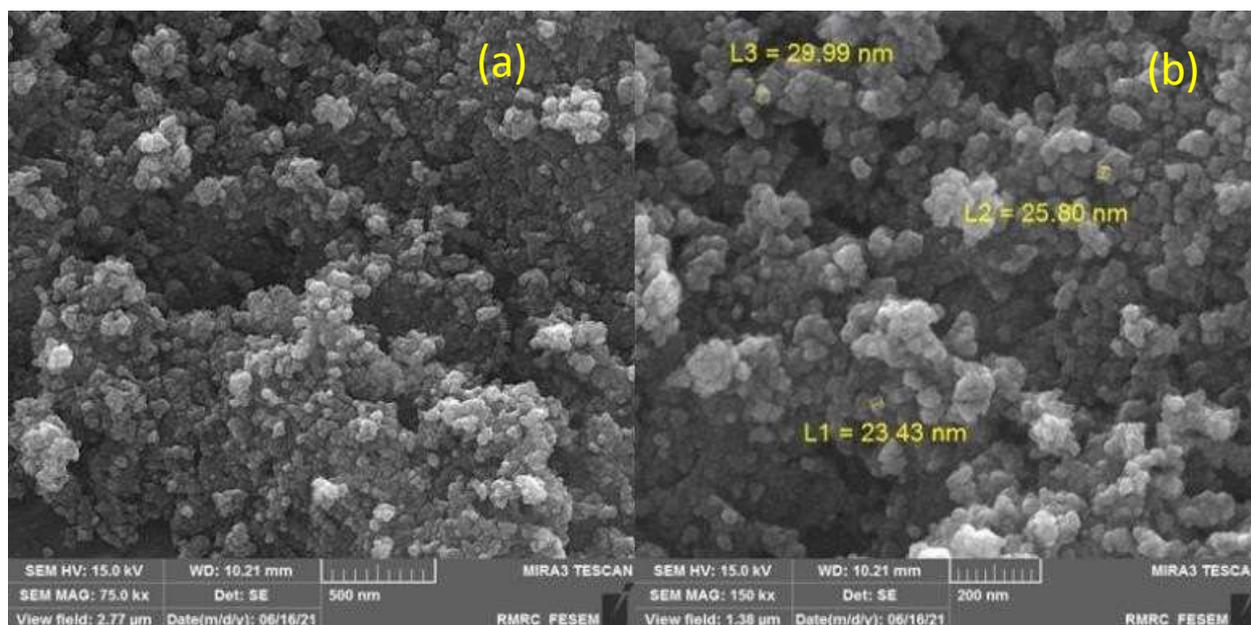


Figure 4 – SEM images of the synthesized nanoparticles.

Figure 5 shows the EDS spectrum of the sample synthesized by the pulsed laser ablation method. The obtained spectrum clearly confirms the presence of iron (Fe) and oxygen (O) elements in the synthesized sample.

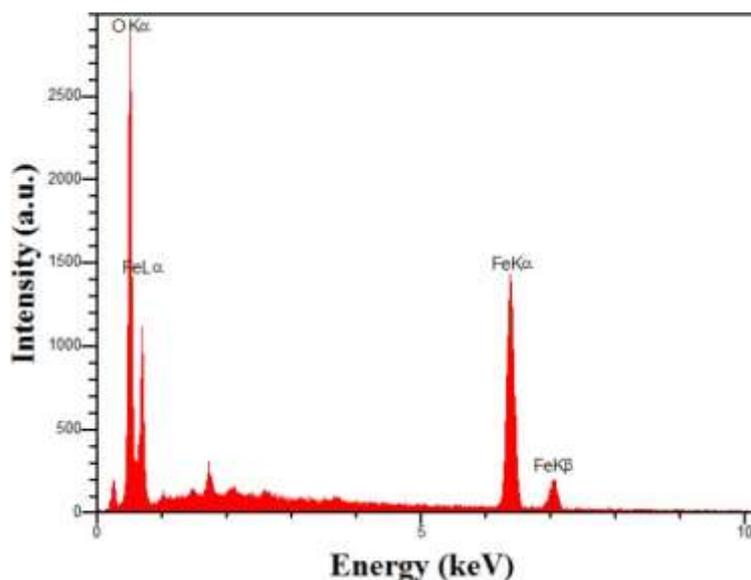


Figure 5 – EDS spectrum of the synthesized sample.

The magnetic behavior of the nanorods was investigated by measuring the magnetization of the synthesized sample using VSM analysis. Figure 6 shows the magnetic curve of Fe₃O₄ nanorods as a function of the applied magnetic field, also referred to as the

hysteresis loop of the iron oxide nanorods. The values of saturation magnetization, remanent magnetization, and coercivity were determined to be 28 emu/g, 0.25 emu/g, and 0.023 kOe, respectively.

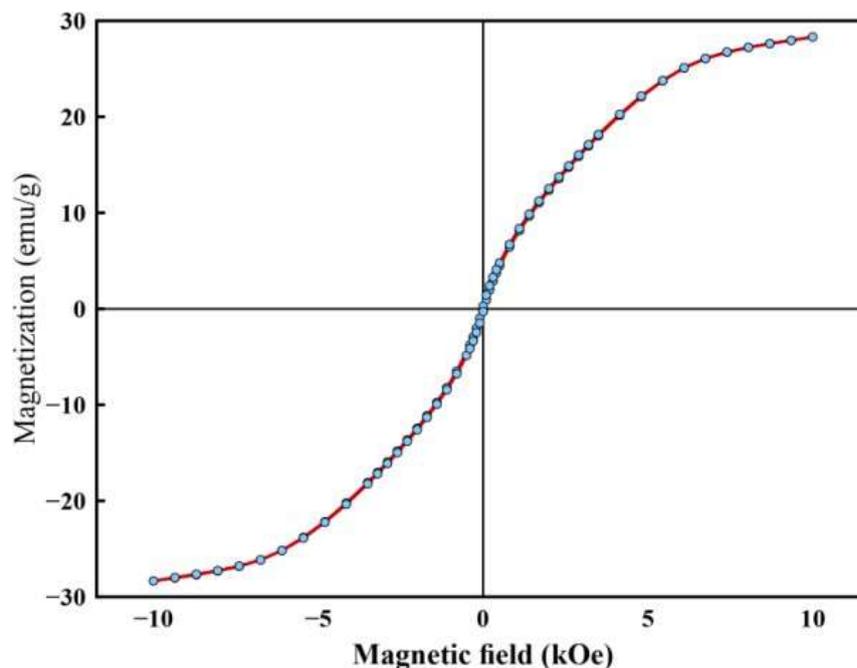


Figure 6 – VSM analysis of the synthesized sample.

4. CONCLUSION

Pulsed laser ablation is a clean and physical method, and the Nano rods synthesized by this technique exhibit high purity. Moreover, the shape and structure of the Nano rods can be controlled by adjusting laser parameters such as power and scanning speed. In this study, the possibility of synthesizing Fe_3O_4 magnetic Nano rods via pulsed laser ablation in a liquid medium (deionized water) was investigated. The XRD pattern of the sample perfectly matched the reference spectrum of Fe_3O_4 Nano rods. The morphology of the Nano rods was analyzed using SEM, which showed that the synthesized Nano rods are spherical in shape. Magnetic characterization of the sample revealed that the saturation magnetization of the synthesized Nano rods is 28 emu/g. The FTIR spectrum clearly exhibited vibrations corresponding to the Fe-O bond in the Nano rods.

The Nano rods synthesized by this method can be directly used in medicine. They can also be utilized as fillers in various matrices such as polymeric, ceramic, and carbon matrices. The Nano rods synthesized through this method can be employed in the removal of industrial pollutants and organic dyes like methylene blue and methyl orange. Furthermore, the produced magnetic Nano rods can be used in the construction of electromagnetic wave shields, sensors, and fuel cells.

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