Synthesis of Magnetite Nanoparticles (Fe₃O₄) Based on Taman River Sand Magnetic Minerals

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Article Info

Abstract

This research aims to produce Fe₃O₄ magnetic nanoparticles-based Taman River sand as the raw material through coprecipitation. The Fe content percentage in the Taman River sand after separation was characterized using XRF testing. The lattice parameters and crystal size of the samples were tested using XRD. The nanoparticles' morphology, structure, particle shape, and elemental content were characterized using SEM-EDX. The magnetic properties and magnetic saturation values were tested using VSM. XRF testing results on the iron sand showed a Fe content of 84.72%. The synthesis resulted in a brNownish-black Fe₃O₄ nanoparticle powder that a permanent magnet could attract. Based on the XRD results, the Fe₃O₄ nanoparticle powder peaks were obtained at X-ray diffraction pattern angles of 2θ: 21.44°, 35.38°, 41.74°, 50.78°, 63.36°, 67.70°, and 74.66°, indicating the presence of Fe₃O₄ nanoparticles. The crystal structure formed was cubic with lattice parameters a = b = c = 8.331 Å and a crystal size of 18.43 nm. SEM-EDX results showed an average particle size of 25-30 nm. VSM results indicated that the Fe₃O₄ sample had a saturation magnetization (Ms) of 27.36 emu/g, a remanent magnetization (Mr) of -0.01 emu/g, and a coercive field (Hc) of 0.01 T.

Introduction

Magnetite nanoparticles (Fe₃O₄) have garnered significant attention in materials science and technology due to their unique magnetic properties, biocompatibility, and diverse potential applications in biomedical technology, electronics, and environmental sectors [1]. Synthesizing Fe₃O₄ from natural materials, such as river sand, is a relevant and growing topic because these resources are abundant and environmentally friendly [2]. Fe₃O₄ nanoparticles have exceptional magnetic properties, including high magnetism and large saturation magnetization [3]. Moreover, these nanoparticles can be easily modified and manipulated, making them highly useful for various applications, ranging from medical to environmental fields [4]. In the medical

field, Fe_3O_4 nanoparticles have been used as contrast agents in magnetic resonance imaging (MRI), agents in cancer therapy through hyperthermia, and targeted drug delivery carriers [5]. The strong magnetic properties of Fe_3O_4 nanoparticles enable precise control and efficiency in these applications [6]. Additionally, these nanoparticles are used for water treatment and pollutant removal in the environmental field, thanks to their ability to adsorb and bind harmful substances [7].

Several studies have been conducted to enhance the effectiveness and efficiency of magnetic materials. Research by Febriyani et al. successfully synthesized Fe_3O_4 from iron ore, observing that the crystal size was 18.1 nm and the crystal structure was cubic [8]. Another study by Prasetrowati et al. synthesized Fe_3O_4 with varying concentrations of NH_4OH. VSM testing results showed that sample 2 (15% NH_4OH) had a saturation magnetization (M_s) of 25.7 emu/g, remanent magnetization (M_r) of 0.06 emu/g, and coercivity (H_c) of 0.023 T. In contrast, sample 4 (25% NH_4OH) had a saturation magnetization (M_s) of 23.6 emu/g, remanent magnetization (M_r) of 2.02 emu/g, and coercivity (Hc) of 0.019 T. Both samples were classified as soft magnetic materials [9].

The methods and parameters of synthesis play a crucial role in determining the size, shape, morphology, crystallinity, and polydispersity of the Fe_3O_4 nanoparticles. These parameters directly affect the physical and chemical properties of the Fe_3O_4 nanoparticles. Several methods for synthesizing Fe_3O_4 nanoparticles include the sol-gel, coprecipitation, and thermal decomposition [10]. In this study, the researchers used the Coprecipitation Method. The coprecipitation method is the simplest, most economical, and most efficient way to obtain Fe_3O_4 powder from natural iron sand. The particles produced by this method are relatively stable and highly insoluble in solvents [9], [10], [11]

Iron sand contains iron particles like magnetite found along rivers and beaches. These deposits are formed through the processes of weathering, erosion by surface water, and wave action that breaks down the parent rock. These particles then accumulate and are washed by sea waves [9]. One river with iron sand deposits is the Taman River. The Taman River is located in Banjar Taman, Batuagung Village, Jembrana District, Bali. The Taman River is often a site for sand mining due to the abundance of river sand deposits. The deposits include not only typical river sand but also black sand deposits. Miners usually take only the regular river sand, ignoring the black river sand deposits, unaware of their value. This black sand is iron sand carried by the river's current towards the sea. Iron sand deposits can contain magnetic minerals such as magnetite (Fe_3O_4), hematite (α-Fe_2O_3), and maghemite (γ-Fe_2O_3) [12].

Based on the above, this research's primary focus is synthesizing Fe_3O_4 nanoparticles using raw materials from Taman River sand. Utilizing river sand as a raw material for nanoparticle synthesis is an innovative and sustainable approach, considering its abundant availability and low cost [13]. This method also offers a solution for optimizing local resources that have not been fully utilized [14]. The research aims to identify the minerals in the Taman River iron sand by synthesizing Fe_3O_4 using the coprecipitation method. After separation, the Taman River iron sand will be analyzed using X-ray fluorescence (XRF). Subsequently, the synthesized product will be characterized using X-ray diffraction (XRD) to determine the parameters and crystal size. The morphology and elemental composition of the synthesized Fe_3O_4 will be examined using a Scanning Electron Microscope with Energy Dispersive X-ray Analysis (SEM-EDX). A Vibrating Sample Magnetometer (VSM) test will also be conducted to evaluate its magnetic properties.

Experimental Method

This study presents a comprehensive experimental investigation focused on developing, synthesizing, and characterizing materials. Fe_3O_4 nanoparticles are synthesized using the coprecipitation method, with Taman River iron sand as the primary raw material. NH_4OH (*Merck*) and HCl (*Merck*) are employed without additional purification, while distilled water is utilized throughout the experimental procedures.

Initially, the iron sand is separated from impurities using a permanent magnet. Subsequently, $FeCl_2$ and $FeCl_3$ are prepared by dissolving the iron sand in 58 mL of HCl and stirring the mixture at 450 rpm for 1 hour.

The synthesis of Fe_3O_4 follows, where 18 mL of $FeCl_2$ and $FeCl_3$ solution is titrated with 25 mL of NH_4OH under magnetic stirring at 450 rpm for 60 minutes. The resulting Fe_3O_4 exhibits acidic properties, necessitating repeated washing with distilled water to neutralize the pH. The Fe_3O_4 is then subjected to heating at 100°C for 1 hour.

After separation, the iron sand is characterized using X-ray fluorescence (XRF) to determine the elemental composition of the iron sand. Then, after the synthesis process is complete, the characteristics of the Fe_3O_4 nanoparticles are tested using X-ray diffraction (XRD) to determine crystal size and lattice parameters, scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX) to assess morphology, structure, particle shape, and elemental content, and vibrating sample magnetometer (VSM) to determine magnetic properties. The entire process is shown in Figure 1.

Result and Discussion

The XRF test results from the iron sand of the Taman River can be seen in Table 1.

Table 1. XRF Test Results Iron Taman River Sand

Element	Percentage (%)
Al	1.60
Si	1.60
P	0.28
Ca	0.90
Ti	7.83
V	0.69
Cr	0.11
Mn	0.50
Fe	84.72
Zn	0.07
Eu	0.59
Re	0.30

The XRF test findings indicate that iron sand contains trace amounts of other component elements. Fe, Ti, and Mn are transition elements that possess magnetic characteristics. Components such as Si, Al, Bi, Re, and other oxide minerals are impurities or nonmagnetic. The XRF results also show a percentage of Fe element of 84.72% after separation. By contrast, Sirua et al. researched iron sand from the Maosu River and got a Fe element percentage of 98.34% by XRF test findings following separation [15]. In addition, Sihombing et al. conducted research that yielded a Fe element percentage of 51.533% using XRF testing after separation. Similarly, Tiwow

et al. found that iron sand from Bontokanang village had a Fe element percentage of 66.7%, while iron sand at Tanjung bayang beach had a Fe element percentage of 79.56%. A greater Fe rate is advantageous for synthesizing Fe_3O_4 nanoparticles [16], [17].
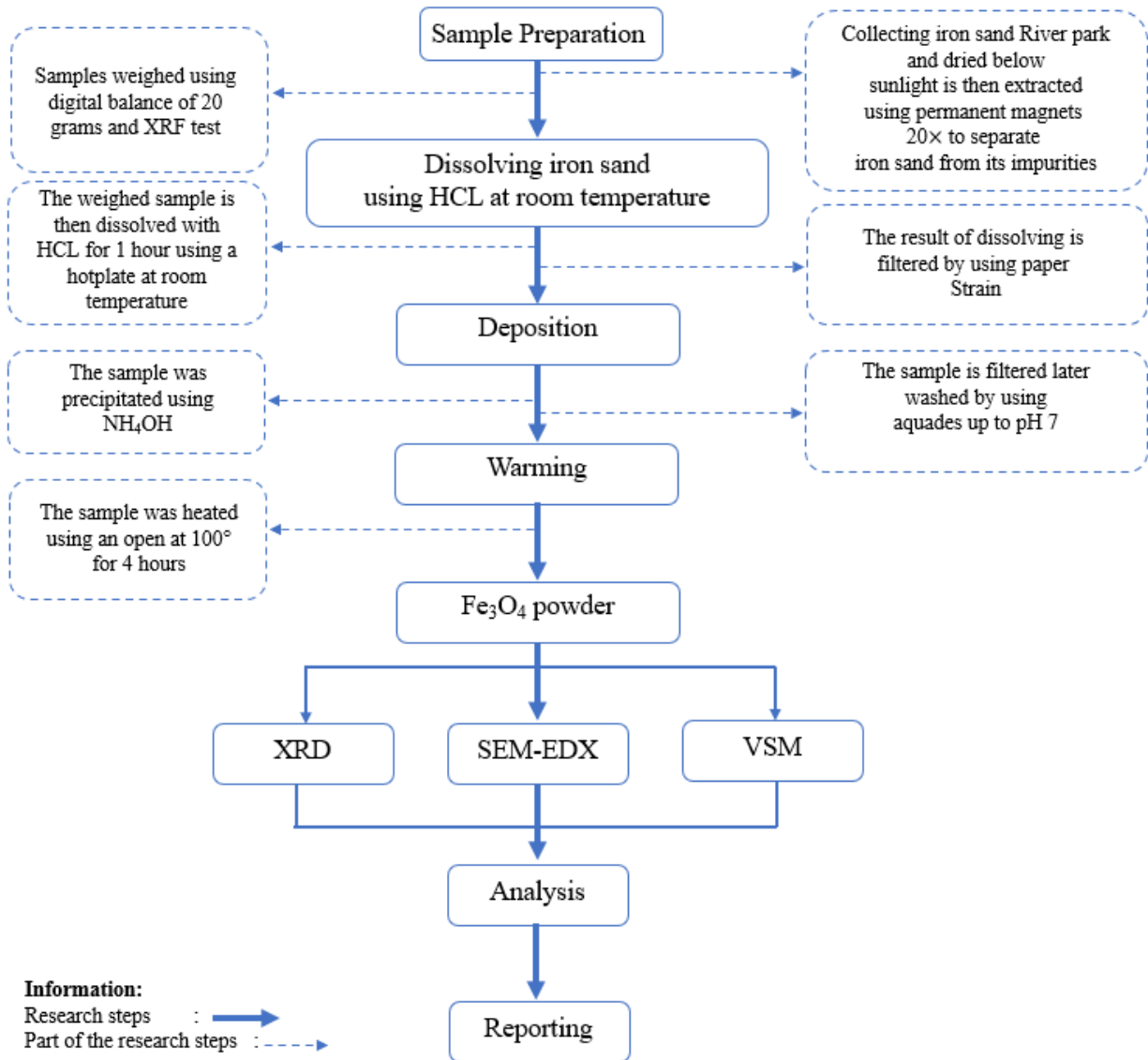
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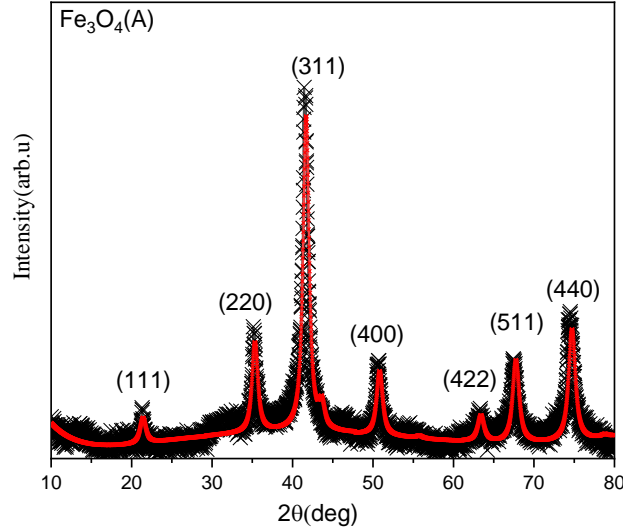

Figure 1. Research flow diagram

Figure 2. X-ray diffraction pattern of Fe_3O_4

The X-ray diffraction pattern presents characteristic peaks of red Fe_3O_4 nanoparticles at 2θ angles: 21.44°, 35.38°, 41.74°, 50.78°, 63.36°, 67.70°, and 74.66°, corresponding to the hkl planes (111), (220), (311), (400), (422), (511), and (440) as per AMCSD No. 0007423 for Fe_3O_4 (Magnetite). The diffractogram pattern of bare Fe_3O_4, especially the HKL 311 plane, shows significant differences from Swastika et al.'s results, which observed Fe_3O_4 nanoparticle peaks at 2θ: 30°, 35°, 43°, 54°, and 63° with hkl planes (220), (311), (400), (511), and (440) [18]. This discrepancy may be due to the oxidation of Fe_3O_4 nanoparticles forming γ-Fe_2O_3 at room temperature. Oxidation can occur due to oxygen from air or dissolved in water.

Based on the quantitative analysis using the Scherrer equation, the crystallite size of Fe_3O_4 was 18.43 nm with a lattice parameter of a=b=c=8.331Å. This finding is supported by Jesus et al., who obtained a lattice parameter of a=b=c=8.348Å, and Swastika et al., who reported a crystallite size of 72 nm. In contrast, Bukit et al. found a crystallite size of 14.90 nm and a lattice parameter of 8.383Å [18],[19],[20].

Table 2. Comparison of crystal size and lattice parameters

Crystal size (nm)	Lattice parameters ($a = b = c$)	Reference
18.43	8.311Å	[18]
72.00	8.348Å	[19] [20]
14.90	8.383Å	

The crystallite size of Fe_3O_4 nanoparticles of 18.43 nm indicates tiny magnetite grains. This nanometric size provides a large surface area, enhancing chemical reactivity and potential applications in various fields such as catalysis, biomedicine, and wastewater treatment. The small crystal size allows better interaction with other materials, thus improving efficiency in these applications [21]. The lattice parameter of magnetite at 8.331 Å describes the distance

between atomic centers in its crystal structure. Changes in lattice parameters can affect magnetite nanoparticles' physical and chemical properties [22].

SEM was used to observe the particle morphology, as shown in Figure 3. The figure shows that Fe_3O_4 nanoparticles have a spherical surface shape with unequal diameters due to aggregation. Additionally, [23]'s research produced spherical morphology. The particle size distribution was analyzed using a Gaussian fit, yielding 25 and 30 nm particle sizes. The synthesis procedure yielded sizes that are consistent with those found in a study by Packiasamy et al., which found nanoparticle sizes ranging from 20 to 100 nm [24].
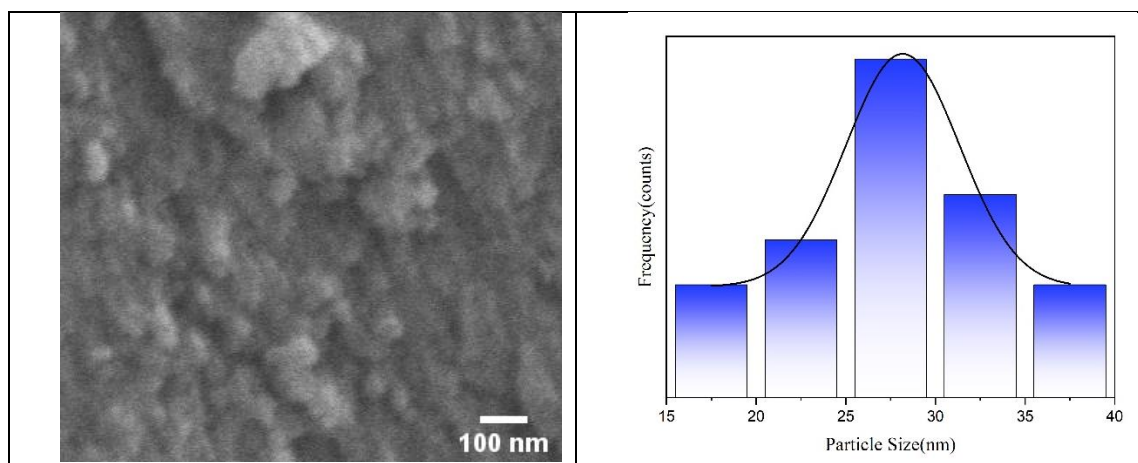
Figure 3. Morphology and particle size distribution of Fe_3O_4

The EDX spectrum results are displayed in Figure 3, indicating that the predominant elements in the nanoparticles are Fe and O. The element percentages within the nanoparticles are illustrated in Figure 4, with Fe at 51.79% and O at 25.68%, confirming the successful synthesis of Fe_3O_4 nanoparticles. These EDX findings are consistent with those of Kurnia et al., who reported Fe at 65.86% and O at 19.18% [25].

The results from EDX also indicate the presence of impurities within the Fe_3O_4 nanoparticles. The existence of these impurities could be caused by several factors. One of them is environmental contamination. The environment in which the synthesis is conducted can serve as a source of contamination. Airborne particles or those near the workspace, dust, or other contaminants can end up in the final product [22].

VSM was used to evaluate the magnetic properties of Fe_3O_4 nanoparticles, as shown in Figure 5. These nanoparticles show type S behavior in their hysteresis curve, which saturates below the applied magnetic field. The hysteresis curve helps depict the relationship between magnetization (M) and external magnetic field (H). Several essential parameters evaluated from the hysteresis curve to determine magnetic properties include saturation magnetization (Ms), coercive field (Hc), and remanent magnetization (Mr) [26]. Whereas Ms is the magnetization value at which all magnetic moments coincide, Hc is the magnetic field intensity required to cancel out magnetization. However, Mr indicates the magnetization that

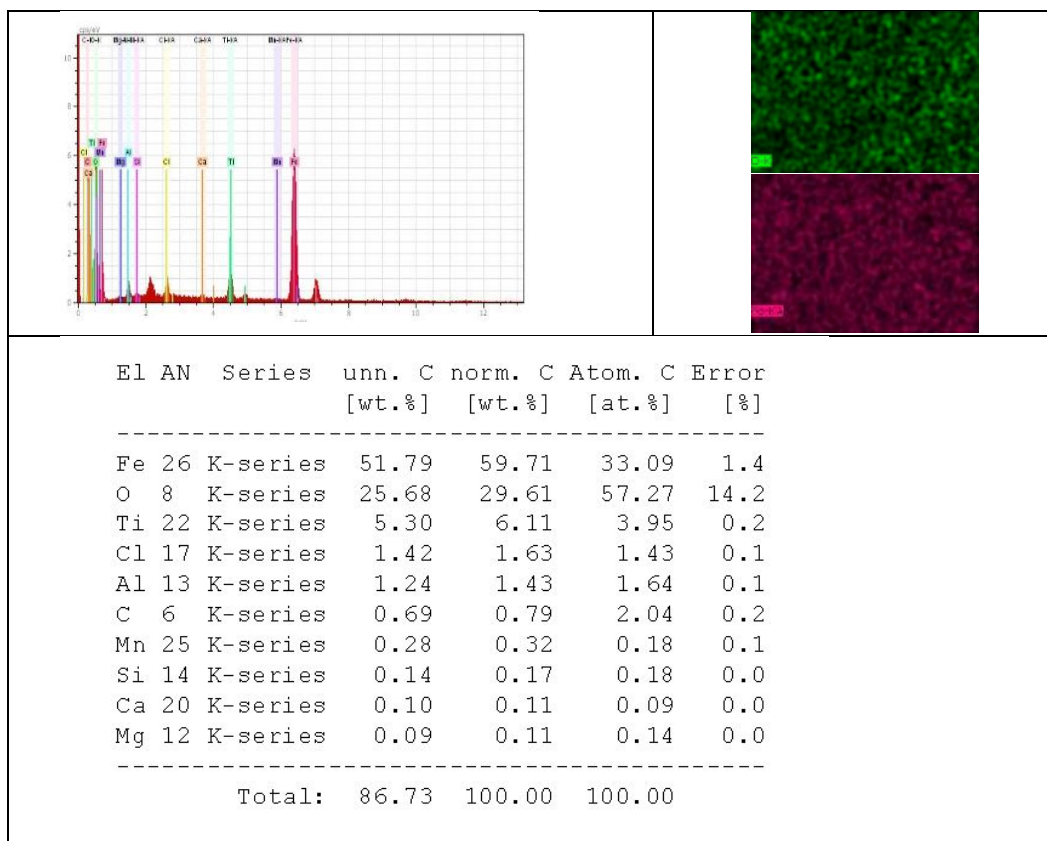remains when the magnetic field is removed. The origin2023 software was used to analyze the gathered VSM data.


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El AN  Series   unn. C norm. C Atom. C Error
                 [wt.%]  [wt.%]  [at.%]   [%]
-------------------------------------------
Fe 26 K-series   51.79   59.71   33.09   1.4
O  8  K-series   25.68   29.61   57.27  14.2
Ti 22 K-series    5.30    6.11    3.95   0.2
Cl 17 K-series    1.42    1.63    1.43   0.1
Al 13 K-series    1.24    1.43    1.64   0.1
C  6  K-series    0.69    0.79    2.04   0.2
Mn 25 K-series    0.28    0.32    0.18   0.1
Si 14 K-series    0.14    0.17    0.18   0.0
Ca 20 K-series    0.10    0.11    0.09   0.0
Mg 12 K-series    0.09    0.11    0.14   0.0
-------------------------------------------
      Total:     86.73  100.00  100.00
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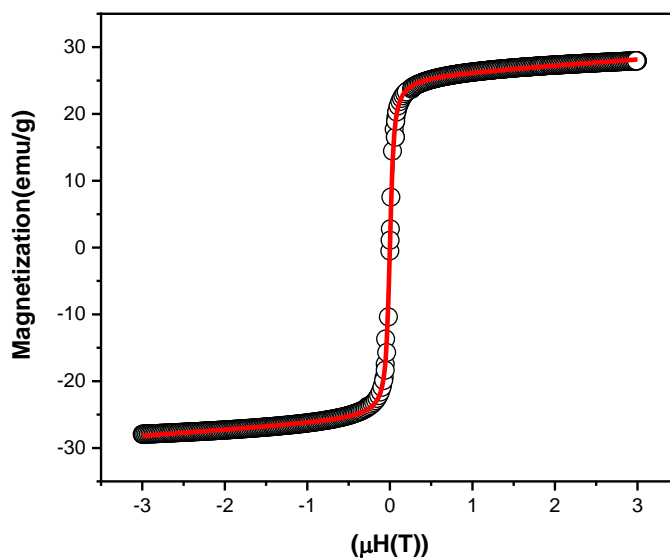**Figure 4.** EDX results of Fe_3O_4 nanoparticles

Figure 5. Hysteresis curve of Fe_3O_4 nanoparticles

According to Figure 5, Fe_3O_4 nanoparticles demonstrate an Ms value of 27.36 emu/g, an Ms value of -0.01 emu/g, and a coercivity of 0.01 T, suggesting their superparamagnetic nature. A study conducted by Prasetyowati et al. reported a Ms value of 25.7 emu/g, a Mr value of 0.06 emu/g, and a Hc value of 0.01 T. Based on its low magnetic coercivity value (0.01 T), the sample can be categorized as a soft magnetic material [9]. This indicates that Fe_3O_4 nanoparticles require a relatively weak magnetic field to change their magnetization direction. Additionally, the magnetic properties of these nanoparticles are heavily influenced by various structural aspects such as size, shape, crystallinity level, and surface properties.

Particle size significantly affects the magnetic properties of the remanent and magnetic saturation of magnetite (Fe_3O_4). The smaller the particle size, the larger its specific surface area, which increases the magnetic interaction between particles. This results in changes in magnetic properties such as saturation magnetization (Ms) and remanent magnetization (Mr) [27],[28]. A high Ms value (27.36) indicates the magnetite sample's ability to achieve maximum magnetization when exposed to an external magnetic field. This suggests that these samples have significant potential for applications requiring solid responses to magnetic fields, such as electronics and energy industries. A Mr value close to zero (-0.01) indicates that the magnetite sample has very little residual magnetization after removing the magnetic field. This is desirable in applications requiring quick and effective removable magnetization, such as magnet-based equipment and data storage.

A low Hc value (0.01) indicates that this sample requires a weak magnetic field to change its magnetization direction. Low Hc materials are suitable for applications that require a quick response to external magnetic fields, such as in sensors and navigation devices [29]. Real-world applications: (1) Electronic Industry: Magnetite samples with high Ms can produce electronic components such as transformers, inductors, and magnet-based devices. Their strong response to magnetic fields makes them valuable in energy efficiency and power generation. (2) Data Storage: Materials with low Hc are highly valued in the data storage industry because they enable fast and efficient data storage in devices such as hard disks and solid-state drives (SSDs). The low Mr property helps prevent data loss due to residual magnetization. (3) Automotive Industry: Magnetite is used in speed sensors, direction sensors, and various applications in vehicles. Its responsive characteristics are beneficial in enhancing vehicle performance and safety [24].

With characteristics measured in this magnetite sample, this material has the potential to contribute to various technology applications that utilize its magnetic properties. Further research and experiments may be needed to develop more specific and efficient applications in multiple industries and disciplines [29].

Conclusion

The coprecipitation approach successfully produced Fe_3O_4 nanoparticles from naturally occurring river sand iron ore. According to XRF analysis, the extracted iron sand's Fe concentration was 84.72%. The resulting crystal structure, with crystal lattice parameters of a=b=c= 8.331 Å and crystal size of 18.43 nm, is cubic inverse, according to XRD examination. The SEM-EDX analysis revealed a morphology of spherical nanoparticles, with diameters varying between 25 and 30 nm. The EDX spectrum verified that Fe_3O_4 nanoparticles with a

composition of Fe (51.79%) and O (25.68%) had formed. The Fe_3O_4 sample showed ferromagnetic properties, as shown by VSM tests, which included coercive field (Hc) = 0.01 T, saturation magnetization (Ms) = 27.36 emu/g, and remanent magnetization (Mr) = -0.01 emu/g.

Acknowledgment

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