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Structural Investigations of Stabilized Magnetite Nanoparticles

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Abstract

This work focuses on the synthesis and physicochemical properties of stabilized magnetite nanoparticles (SMNPs) using the eco-friendly agent starch as a stabilizer. Nanoparticles were prepared by the Fe^{2+}/Fe^{3+} ions coprecipitation approach in alkali media (ammonia hydroxide) as a precipitating agent. Relevant properties of the obtained nanoparticles were investigated by a vibrating sample magnetometer (VSM) and dynamic light scattering (DLS). The morphology was monitored by transmission electron microscope (TEM) and scanning electron microscope (SEM). The average crystallite size of SMNPs, about 10 nm, is observed from TEM and DLS. The presence of starch in the synthesis medium lowers the magnetite particle size, increases the dispersion of the particles, and promotes the superparamagnetic nature of MNPs.

Keywords

Magnetite Nanoparticles, Coprecipitation, Ammonia Hydroxide, Starch, Characterization

1. Introduction

Nanotechnology has been regarded as one of the most significant contemporary advances in science and technology. Nanoparticles are the main building components in the production and development of nanomaterials [1]. As a result, nanoparticles have aroused widespread interest among academics because of their unique characteristics, such as form, size, and dispersion, which can be used in a variety of applications. Iron oxide nanoparticles play an important role in many chemical, physical, and material sciences [2]-[5].

Nanoparticles of iron oxide as magnetite (Fe_3O_4) have recently received a lot of attention due to their diverse physical and chemical properties at the nanoscale [6]. The magnetic and electric properties of magnetite (Fe_3O_4) nanoparticles are

very remarkable [7]. The cubic inverse spinel structure is unique, and it might be an n-type or p-type semiconductor. It has minimal resistance among iron oxides due to its small band gap (0.1 eV) [8]. Due to the alternating Fe^{2+}/Fe^{3+} lattice interrupted by oxygen atoms that allows electrostatic interaction, magnetite is ferromagnetic [9].

Moreover, surface alteration of magnetite nanoparticles can lead not only to increased stability in solvents, but also to enhanced interfacial features and avoidance of particle aggregation [10]. Magnetite nanoparticles have exclusive properties, including good magnetic and electric properties, large surface area, broad surface-to-volume ratio, simple separation under an external magnetic field, catalytic activity, respectable chemical activity, susceptibility to oxidation, biocompatibility, and low toxicity properties [11].

These iron oxides find applications as catalysts [12], adsorbents [13], pigments [14], flocculants [15], coatings [16], gas sensors [17] [18], wastewater treatment [19] [20], and for lubrication [21] [22]. Likewise, these nanoparticles can also be used in various advanced processes to form nanoreactors and to be added to polymer films and other products based on their superior magnetic properties [23]. For these applications, superparamagnetic nanoparticles are made from iron oxide, and there is a need to develop and modify these synthesis processes with minimal operational cost.

There are several diverse methods to synthesize magnetite nanoparticles, such as the coprecipitation method [24], sol-gel method [25], oxidation method [26], reduction method [27], hydrothermal method [28], solvothermal method [29], thermal decomposition method [30], and microwave-assisted synthesis [31]. The coprecipitation approach in aqueous media is a suitable method for magnetite synthesis since the synthesis process is easy and the environmental impact is minimal.

The coprecipitation method is a simple way to make magnetite nanoparticles from an aqueous iron salt (Fe^{2+}/Fe^{3+}) solution by adding a base in an inert environment. The coprecipitation technique produces no hazardous intermediates or solvents, requires no precursor complexes, and operates at temperatures below 100˚C. Due to its ability to expand, reproduce, and environmentally acceptable reaction conditions, this technology has been recognized for its industrial utility [32].

In this work, magnetite nanoparticles were successfully prepared through the Fe^{2+}/Fe^{3+} ions coprecipitation approach. Controllable preparation of magnetite using starch as a stabilizer and ammonia hydroxide as a precipitating agent was studied. The effect of starch with the precipitating agent on morphology, particle size, dispersion, and the superparamagnetic nature of the as-prepared magnetite nanoparticles was investigated. Various tools have been used to investigate the prepared magnetite nanoparticles, such as transmission electron microscope (TEM), scanning electron microscope (SEM), vibrating sample magnetometer (VSM), and dynamic light scattering (DLS).

2. Experimental

2.1. Materials and Methods

2.1.1. Materials

All chemicals were analytical grade reagents, obtained commercially and used as received without further purification. Ferric chloride ($FeCl_3 \cdot 6H_2O$), ferrous sulphate ($FeSO_4 \cdot 7H_2O$), starch, and ammonium hydroxide solution were used. Distilled water and deionized water were used throughout the experiment for preparing solutions and washing purposes.

2.1.2. Synthesis of Magnetite Nanoparticles (MNPs)

MNPs were synthesized using ferrous ions (Fe^{+2}) and ferric ions (Fe^{+3}) as precursor salts and starch as a capping agent. The preparation method starts with the addition of stock solutions of ferrous ions (Fe^{+2}; 1 M)/ferric ions (Fe^{+3}; 2 M) to 100 ml of starch (1.2%) solution and stirring for 30 min. Stock solutions were mixed and heated at 80˚C, and then ammonia hydroxide solution (30%) was introduced by syringe dropwise until the pH was adjusted to around 12. The appearance of black color indicated the formation of magnetite nanoparticles. The black mixture was heated at 80˚C for 60 min, filtered, washed with deionized water repeatedly until the pH became neutral, and dried at room temperature.

To calculate the percentage yield for magnetite nanoparticle synthesis, the actual mass of the dried product is divided by the theoretically calculated mass of the product and multiplied by 100. The theoretical mass is determined by using the limiting reactant from the initial iron salt solutions (1:2 molar ratio) and the stoichiometry of the reaction to find the maximum amount of magnetite that can be formed (obtained in 90% yield).

2.2. Characterization Techniques

The characterizations of MNPs were investigated by a vibrating sample magnetometer (VSM) and dynamic light scattering (DLS). The morphology was monitored by transmission electron microscope (TEM) and scanning electron microscope (SEM).

Transmission Electron Microscope (TEM): The TEM images were taken by a JEM-2100 operated at an accelerating voltage of 200 kV.

Scanning Electron Microscopy (SEM): The morphology of the powder sample of Fe_3O_4 nanoparticles was analyzed using scanning electron microscopy (JEOL SEM, JSM-636OLA, Japan) at an accelerated voltage of 20 kV.

Vibrating Sample Magnetometer (VSM): Magnetic characteristics were measured by VSM (Lake Shore-7410 vibrating sample magnetometer, USA), with a magnetic field up to 30,000 Oe.

Dynamic Light Scattering (DLS): The average size was examined by means of dynamic light scattering (DLS, Zetasizer Nano-ZS, Malvern Instruments, London, UK).

3. Results and Discussion

3.1. Morphological Characterization Studies

TEM Analysis: The morphology and particle size of the prepared MNPs were investigated by transmission electron microscopy. As shown in Figure 1, the MNPs showed a spherical shape with uniform particle size without aggregation (average about 10.39 nm).

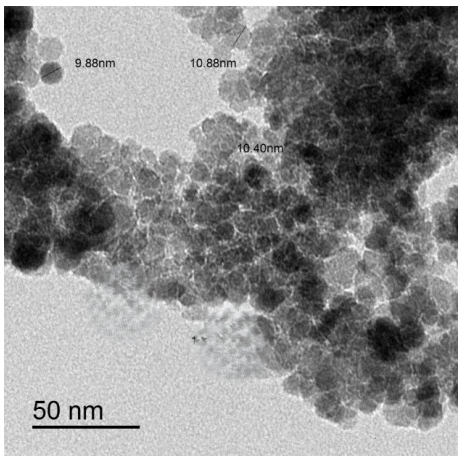

Figure 1. TEM micrograph of MNPs.

SEM Analysis: The SEM micrograph of MNPs is shown in Figure 2. The scanning electron micrograph of MNPs shows smooth particles, and they are slightly spherical in shape.

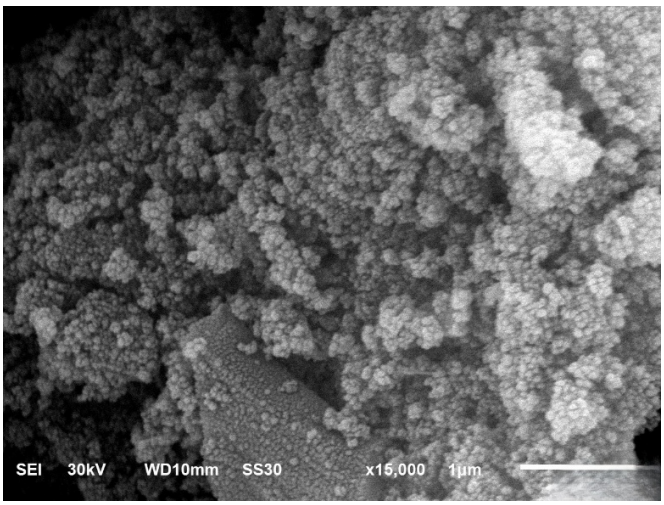

Figure 2. SEM micrograph of MNPs.

3.2. Magnetic Analysis

Vibrating Sample Magnetometer (VSM): Figure 3 represents the room temperature M-H curve [the relation between the applied magnetic field (H; Oe) and the magnetization (M; emu/g)] of magnetite nanoparticles. The magnetic saturation

(Ms) of MNPs shows soft magnetic behavior with a relatively good value of 57.43 emu/g and no hysteresis loop, which means that the superparamagnetic nature of this sample and a negligible coercive value (Hc = 4.19 Oe) indicate the retained superparamagnetic property. The superparamagnetism of MNPs could be a potential candidate for effective applications with recyclable capacity and minimal release into the environment.

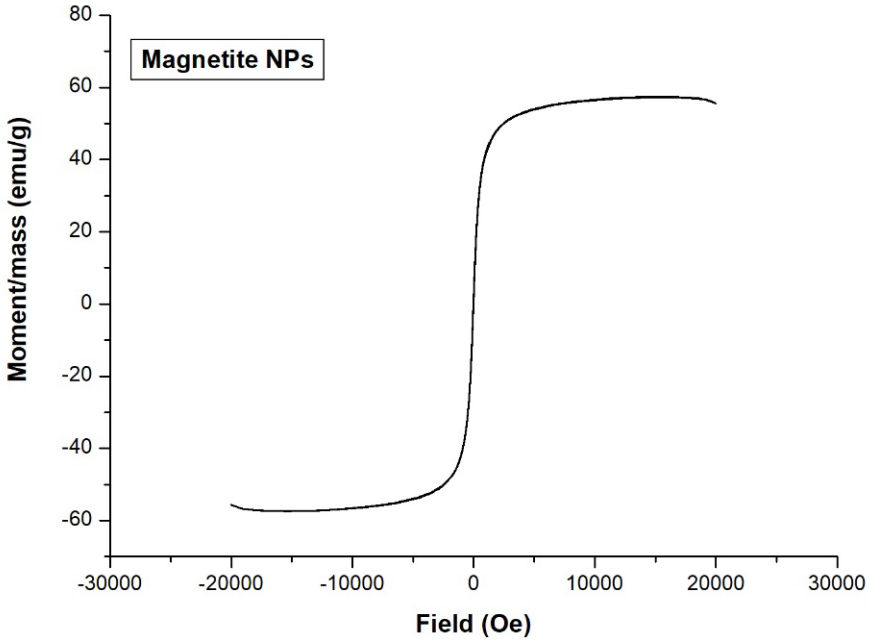

Figure 3. M-H curve of MNPs.

3.3. Size Distribution

Dynamic Light Scattering (DLS): DLS, also known as photon correlation spectroscopy, is one of the most popular methods used to determine the size of nanoparticles. The hydrodynamic radius is the radius of a sphere that has the same diffusion coefficient within the same viscous environment as the particles being measured. It is directly related to the diffusive motion of particles.

Figure 4 shows the distribution of MNPs via dynamic light scattering (decay curve). The average particle size measured at (forward angle 11.1˚) of the synthesized MNPs was 10.60 nm, and the diffusion coefficient value was 4.05×10^{-11} m^2/s. The measurement is consistent with TEM analysis.

By analyzing the decay curve, DLS determined the average particle size and showed that fast decay indicates fast-moving, smaller particles. The short size distribution can indicate good stability and dispersion of MNPs. In addition, it indicates good physical contact between target materials and hybrid nanoparticles, which are beneficial for their performance in applications.

In magnetite synthesis, starch acts as an eco-friendly functionalizing agent that helps control the size and dispersion of magnetite nanoparticles, promoting smaller, more dispersed crystallites with enhanced superparamagnetic properties, com-

pared to the previous study without using starch [33]. Starch chains may provide nucleation points for iron hydrolysis and particle growth, preventing agglomeration and creating stable particles. This functionality can be achieved through direct chemical and hydrogen bonds between starch hydroxyl groups and the iron atoms on the magnetite surface.

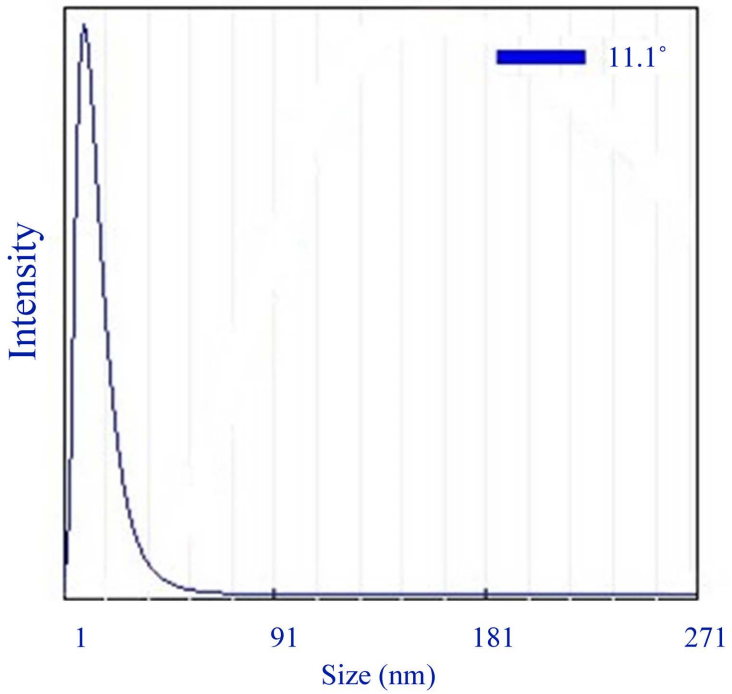

Figure 4. Dynamic light scattering (decay curve) of MNPs.

4. Conclusion

In the current work, magnetite (Fe_3O_4) nanoparticles were controllably prepared in good yield by the coprecipitation method at a relatively lower temperature using starch as a stabilizer and ammonia hydroxide as a precipitating agent. The results revealed that ammonia hydroxide in the presence of starch results in obtaining magnetite nanoparticles with a small particle size, good saturation magnetization value, and increased dispersion of the particles. This preparation approach could highlight the synthesis of stable magnetite nanoparticles for various influential applications.

Conflicts of Interest

The author declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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