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Characterization of Supermagnetic Magnetite Powder Synthesized with Water Extracts of *Moringa oleifera* Leaves and $FeCl_2 \cdot 7H_2O$

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Abstract: The surface coated magnetite nanoparticles dispersed into water can be used in clinical for magnetic resonance imaging, for medical diagnosis and for magnetic field-assisted cancer therapy. Based on their applications in many fields, several methods are widely used and reported in literature for the synthesis of iron oxide nanoparticles. In the present paper, we report on the green synthesis of magnetite phase of iron oxide nanoparticles (Fe_3O_4), which were fabricated using water extracts from the leaves of *Moringa oleifera* and the inorganic salt $FeCl_2 \cdot 7H_2O$, as iron precursor. The obtained powder was characterized through spectroscopic and thermal methods. The vibrating sample magnetometer was used in order to find out about the magnetic properties of the prepared sample. The Fourier transform infrared spectra (FT-IR) have shown peaks at 402 cm^{-1} and 593 cm^{-1}, thus confirming the presence of magnetite in powder. X-ray diffraction gave peaks confirming the presence of Fe_3O_4 in its magnetite phase. Electronic transmission microscopy had indicated that the crystals obtained was of a spherical shape with an average diameter of 50 nm. Gravimetric thermal analysis (GTA) showed a peak centered at around 332°C, signalizing the thermal decomposition of the magnetite. The magnetic properties of the prepared powder exhibited the measured lower coercivity and remanence, demonstrating that the powder under study was made of superparamagnetic particles, suggesting that the prepared magnetite could be a possible candidate for biomedical applications.

Keywords: *Moringa oleifera*, Magnetite, Supermagnetic, Green Synthesis

1. Introduction

Nanomaterials have, for some time now, raised a steadily growing interest in research because of the particular properties they display, as compared to the properties of corresponding materials of macroscopic size. Diverse synthesis procedures have been worked out and have yielded nanomaterials characterized by different structures, size and properties (e.g. optical, magnetic, etc.). Nowadays, nanomaterials are used in areas as diverse as medicine, catalysis, communications, etc. [1].

The various iron oxides, iron (II) oxide (*FeO*), hematite (α-Fe_2O_3), maghemite (γ-Fe_2O_3) and mixed iron (II) and iron (III) oxide, magnetite (Fe_3O_4), have also been transformed into corresponding nanoparticles and subsequently used in many areas of application. Magnetite (Fe_3O_4) is widely known to be a mineral displaying a very pronounced magnetic property [2].

This has consequently prompted a particular interest from researchers who have developed several synthesis methods to produce nanoparticles of Fe_3O_4. Out of these methods, the following can be mentioned: Co-precipitation method [3, 4],

sol-gel method [5], hydrothermal synthesis [6-8], solid state synthesis [9], flame synthesis [10], thermal decomposition [11], solvothermal [12] and by gamma irradiation [13], etc.

However, most of the synthesis methods, physical as well as chemical, of these iron nanoparticles present a number of drawbacks [14] such as high costs for their production, toxicity of the used chemicals, and release of dangerous by-products. In spite of the information gathered so far, we have focused our attention on magnetite with the aim of using an ecological pathway towards its production. The purpose of this paper is the synthesis of magnetite, Fe_3O_4, in a different way, using $FeCl_2 \cdot 7H_2O$ in the presence of water extracts from *Moringa oleifera*, thus challenging the previous methods employed so far. Attempts to characterize the nanoproducts obtained will be made.

2. Materials and Methods

2.1. Materials

The leaves of *Moringa oleifera* were collected in the experimental garden of the Biology Department, University of Kinshasa, Democratic Republic of the Congo (DRC). They were dried for 11 days in the shade to avoid any photochemical degradation from the sun. $FeCl_2 \cdot 7H_2O$ with 99% purity and bidistilled water were supplied from Belgium.

2.2. Preparation of Water Extract

100 g of the powder of *Moringa oleifera* leaves were mixed under stirring with 1000 mL of bidistilled water and heated at 80°C for 20 minutes. The water extracts were recovered from the mixture after filtration.

2.3. Synthesis of Supermagnetic Magnetite Nanocrystals

An aliquot of 20 mL of water extract were mixed under stirring with 100 mL of $FeCl_2 \cdot 7H_2O$ (0.8 M) for 45 minutes, and then kept in darkness for 18 h. The solution was afterwards heated for 20 h at 120°C. The powder obtained was purified by washing it several times and kepts for 2 h at 400°C. The resulting powder has served for the characterization.

3. Results and Discussion

3.1. XDR Analysis

An X-ray diffractogrameter (D/MAX-2550, λ = 1.54056 Å) having a nickel filter was used to determine the structure of the synthesized powder. Figure 1 exhibits the principal peaks characterizing reflections at 2θ equals to 15, 30, 35, 43, 60 and 64°. This corresponds respectively to the reticular plans (111), (220), (311), (400), (511) and (440). Comparison with data from the American Society for Testing Materials (ASTM) confirms that our crystals are of cubic structure in the magnetite phase (PDF No: 89-0688). It can be noticed that the peaks are rather broad, very likely because of the reduced size if the nanoparticles [15, 16].

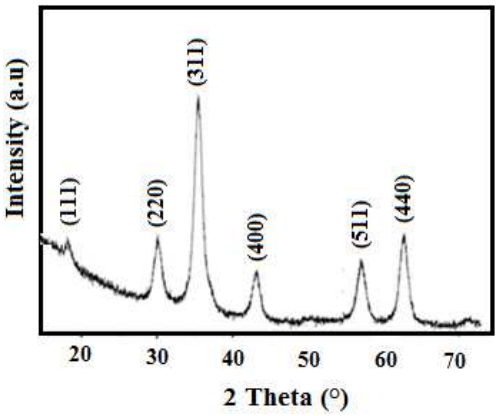

Figure 1. *Diffractogram of mixed iron oxide (magnetite) powder synthesized from extracts of Moringa Oleifera leaves. The various Bragg peaks are indexed by the corresponding Miller indices.*

If the Sherer's equation (1) is used and applied to the plan (311), the average diameter D of the particles making up the powder is found to be 47 nm, close to the value (of 50 nm) obtained through the Electronic Transmission experiment.

$$D = \frac{0{,}92 \times \lambda}{\beta \times cos\theta} \quad (1)$$

In this equation, 0.92, λ, β and θ represent a constant used for particles of spherical morphology, the incident X-ray wavelength, the breadth at half-high of the most intense diffraction peak, and the diffraction angle, respectively.

3.2. FT-IR Analysis

FT-IR spectra were recorded on the sample ground up with KBr. Figure 2 shows peaks at 402 and 593 cm^{-1} attributable to the stretching vibration of Fe-O [17]. The band at 1600 cm^{-1} could originate from distortion vibration of water molecules adsorbed on the surface of magnetite particles. The characterization of the sample of our powder by FT-IR and XRD confirms the presence of magnetite nanoparticles in the sample.

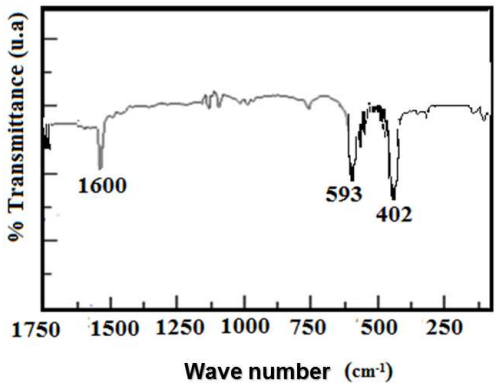

Figure 2. *FT-IR spectrum of the powder prepared from extracts of Moringa Oleifera leaves.*

3.3. TEM Analysis

As can been see in Figure 3, Transmission Electronic

Microscopy revealed that our nanoparticles were spherical in morphology with an average diameter of 50 nm. This diameter is ideal for possible biological applications in the body [2]. The inset in Figure 3 displays a diffraction pattern which is a good proof of the crystallinity of the mixed oxide prepared through our method.

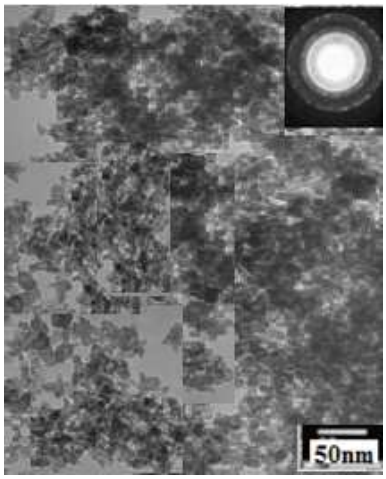

Figure 3. *TEM image of cross section of samples of magnetite nanocrystals synthesized from extracts of Moringa Oleifera leaves. Inset: diffraction pattern obtained from the synthesized powder.*

3.4. Gravimetric Thermal Analysis

The evolution of the mass curve (15 mg) during a rise of the temperature under an inert gas (Argon) was recorded with a heating rate of 10°C/minute and is presented in Figure 4.

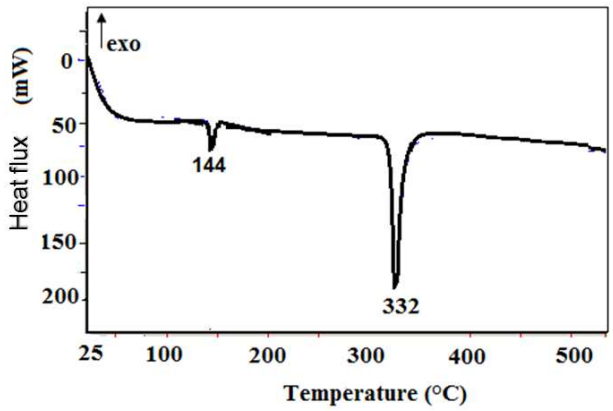

Figure 4. *Curve of the Gravimetric Thermal Analysis of the magnetite powder.*

In Figure 4, Gravimetric Thermal Analysis shows two peaks: a sharp and intense peak at 332°C resulting from the decomposition of magnetite [18] and another peak, much weaker, at 144°C which is very likely due to the evaporation of water adsorbed on the powder surface [19].

3.5. Analysis of Magnetic Properties

Concerning the magnetic properties of our sample, it was noticed, as displayed in Figure 5, that unlike a ferromagnetic behavior characterized by hysteresis loops upon interaction with a magnetic field, the two magnetization curves observed with our sample are very close, thus excluding any hysteresis. Hence, this strongly suggests that our sample is not ferromagnetic but rather supermagnetic.

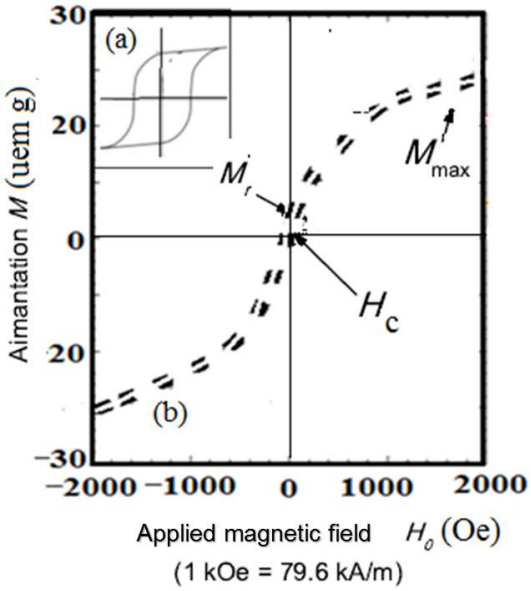

Figure 5. *(a): hysteresis curve of the Fe_3O_4 purchased on the market; (b) Curve measured at T = 300 K for the sample of the magnetite powder produced. M_r corresponds to the residual magnetization, H_c to the coercive magnetic field and M_{max} to the magnetization measured at the maximum magnetic field allowed by the VSM (2 kOe).*

4. Conclusions

The present study has shown that the ecological synthesis (qualified as green) of magnetite nanoparticles (Fe_3O_4) from plant extracts (*Moringa oleifera*) is a good alternative to chemical synthesis, because it is free from pollutants and it protects the environment. The results obtained have showed that the particles turned out to be nearly spherical and of stable shape with a good average diameter which suggests potential applications in the biomedical field. Future research work is envisaged in this direction.

Author Contributions

All the authors have equally contributed to this paper.

Conflicts of Interest

All the authors do not have any possible conflicts of interest.

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