Archives of Materials Science and Engineering

Volume 109
Issue 2
June 2021
Pages 80-85

International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

DOI: 10.5604/01.3001.0015.2627

Synthesis and magnetic properties of Fe_2O_3 nanoparticles for hyperthermia application

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ABSTRACT

Purpose: The main purpose of this publication is to bring closer co-precipitation method of magnetic particles synthesis. Procedure of examining and characterisation of those materials was also shown.

Design/methodology/approach: During the work, the properties and possible biomedical application of the material produced were also examined. Surface morphology studies of the obtained particles were made using Zeiss's Supra 35 scanning electron microscope and S/TEM TITAN 80-300 transmission electron microscope. In order to confirm the chemical composition of observed layers, qualitative tests were performed by means of spectroscopy of scattered X-ray energy using the Energy Dispersive Spectrometer (EDS). The Raman spectra of the samples were measured with a InVia Raman microscope by Renishaw. Magnetic properties of hematite nanoparticles were made using VSM magnetometer.

Findings: Using VSM magnetometer proved that obtained material is mixture of ferromagnetic and superparamagnetic domain.

Practical implications: Magnetic Nanoparticles (MNPs) has been gaining an incrementally increasing interest of scientists in the biomedical areas. Presented materials can be used in the hyperthermia phenomena which can be used in precise cancer treatment.

Originality/value: Specific magnetic properties which determinate obtained material to be well for hyperthermia phenomena.

Keywords: Magnetic nanoparticles, Hyperthermia, Superparamagnetism

Reference to this paper should be given in the following way:

K. Szmajnta, M.M. Szindler, M. Szindler, Synthesis and magnetic properties of Fe_2O_3 nanoparticles for hyperthermia application, Archives of Materials Science and Engineering 109/2 (2021) 80-85. DOI: https://doi.org/10.5604/01.3001.0015.2627

PROPERTIES

1. Introduction

Magnetic nanoparticles are gaining increasing interest from scientists in various fields. Particularly promising are the achievements of research groups dealing with the use of these materials in the diagnosis and eradication of cancer. Magnetic hyperthermia is a phenomenon of local temperature increase due to interaction with a magnetic field. This phenomenon is used both in imaging techniques (MRI – magnetic resonance imaging) and the precise destruction of individual clusters of cancer cells. An important property of nanoparticles for medical applications is their size. They must be adequately small and well dispersed, because after combining into agglomerates they lose their extraordinary properties. It is widely considered that best diameter of such particles is 5-12 nm. Bigger forfeit their extraordinary magnetic properties, smaller – cannot be easily controlled [1-7].

The magnetic properties of materials are determined on the basis of changes in the vector of its magnetic moment in the external magnetic field. On this basis, diamagnetics, paramagnetics, ferromagnetics, anti-ferromagnetics and ferrimagnetics can be distinguished. In paramagnetics, magnetic domains line up parallel to the magnetic field lines, but after the disappearance of the field, the order is destroyed. Paramagnetic materials below a specific temperature, called Curie temperature, become ferromagnetic. This means that even after the disappearance of the external magnetic field, they exhibit residual magnetization, as shown by the hysteresis loop. In nanostructured materials, the role of the entire magnetic domain is taken over by a single particle. If after the disappearance of the external field the average magnetic moment of the nanostructured material is 0, then such material is a superparamagnetic. It causes narrowing of the hysteresis, and sometimes its complete disappearance. Such structures show a strong response to changes in the external magnetic field and a much greater magnetic susceptibility than paramagnetics, which increases their efficiency in cancer therapy applications [8-15].

Research using the VSM magnetometer (Fig. 1) finds wide applications in the magnetization measurements of nanostructured materials. This is due to the possibility of measuring on very small, even microgram, amounts of input material. The sample placed in the device vibrates as a result of interaction with a homogeneous external magnetic field. The device allows testing of materials in the temperature range from 1 to 400 K. The main element of the magnetometer is a set of detectors that use the interference of current carriers to detect and record small changes in magnetic induction. The high homogeneity of the field allows the testing of even weakly magnetic materials [16-18].

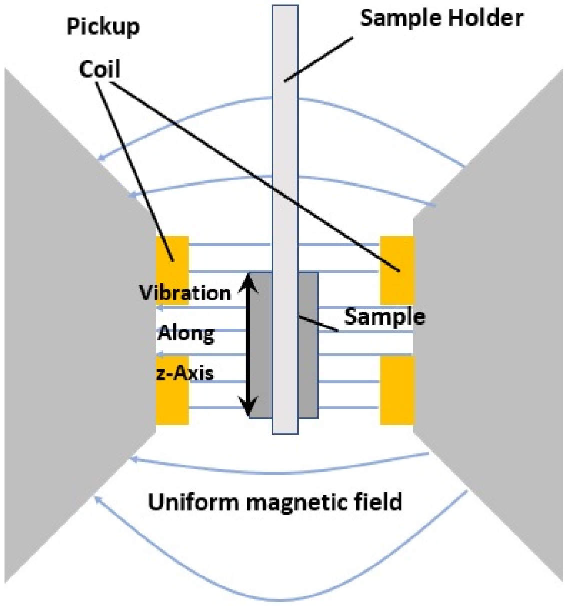

Fig. 1. Scheme of VSM magnetometer probe

As a result of the experiment presented below, the procedure for producing iron (III) oxide nanoparticles is presented. During the work, the properties of the material produced were also examined.

2. Materials and methods

2.1. Synthesis of Fe_2O_3 nanoparticles

The procedure for producing Fe_2O_3 nanopowders (Fig. 2) began by dissolving $FeCl_3$ and $FeCl_2•4H_2O$ in hydrochloric acid. The solution was then vigorously stirred for 2 hours at room temperature, gradually dropwise adding ammonium hydroxide. After this the pH of the solution was about 10. The brown precipitate was then dried for 48 h at 80°C. Then the material was ground using an agate mortar.

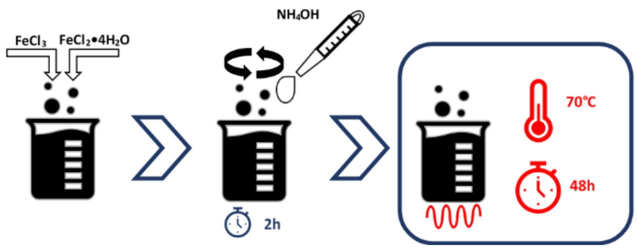

Fig. 2. Scheme of co-precipitation method of Fe_2O_3 nanoparticles

2.2. Structural and magnetic properties examination

Surface morphology studies of the obtained particles were made using Zeiss's Supra 35 scanning electron microscope and S/TEM TITAN 80-300 transmission electron microscope. In order to confirm the chemical composition of observed layers, qualitative tests were performed by means of spectroscopy of scattered X-ray energy using the Energy Dispersive Spectrometer (EDS). The Raman spectra of the samples were measured with a InVia Raman microscope by Renishaw. Magnetic properties of hematite nanoparticles were made using VSM magnetometer.

3. Results

In the SEM imaging (Figs 3, 4) agglomerates of nanoparticles with sizes up to 100 nm were registered.

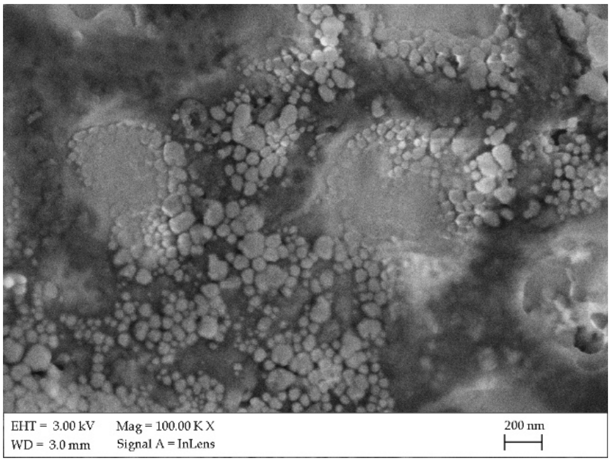

Fig. 3. SEM image of uncoated Fe_2O_3 nanoparticles

The qualitative analysis of EDS (Fig. 5) confirmed the chemical composition of the material produced, no additional contamination of the samples was found. Cu and C on Fe_2O_3 EDS spectre was caused by preparation sample on copper meshwork with carbon membrane. Al mark was caused by holder. TEM imaging (Fig. 6) showed that the size of the nanoparticles did not exceed 10nm. Precipitation in the form of nanowires or nanoflakes (Fig. 7) with a length of about 1 µm was also visible. After that Raman shift (Fig. 8) was examined. Experiment showed that produced nanopowders are mix of hematite (α-Fe_2O_3) and maghemite (γ-Fe_2O_3).

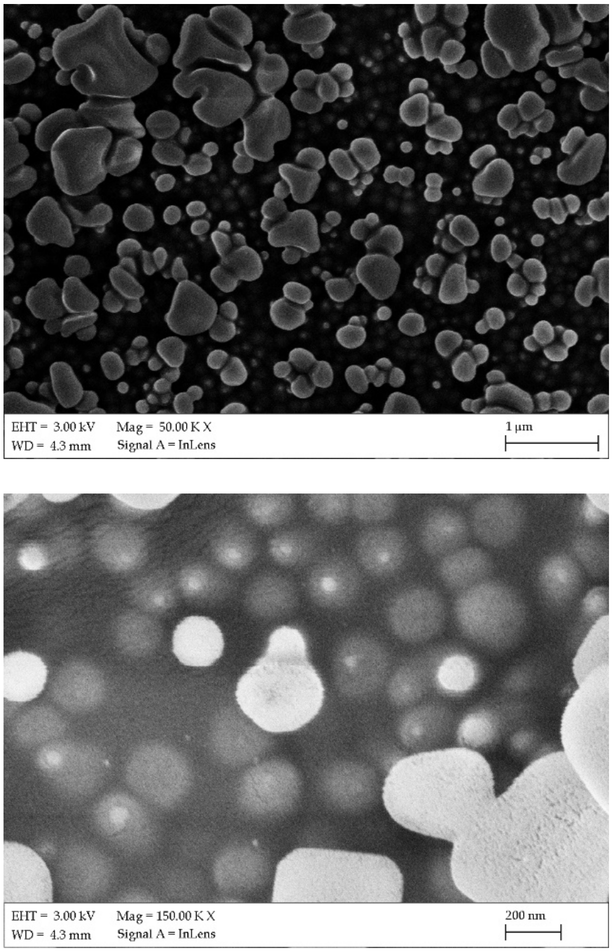

Fig. 4. SEM image of Au-Pt coated nanoparticles

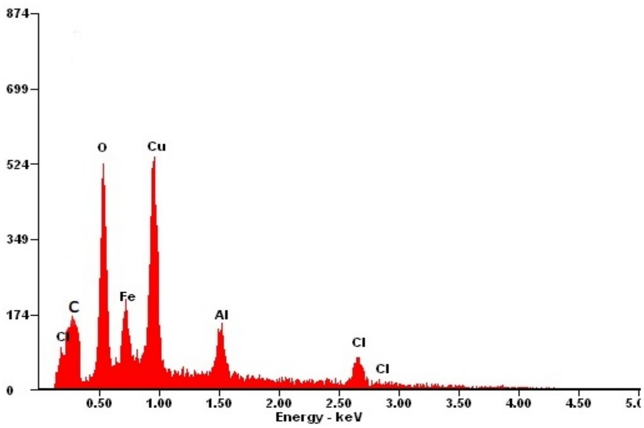

Fig. 5. EDS spectre of Fe_2O_3 nanoparticles

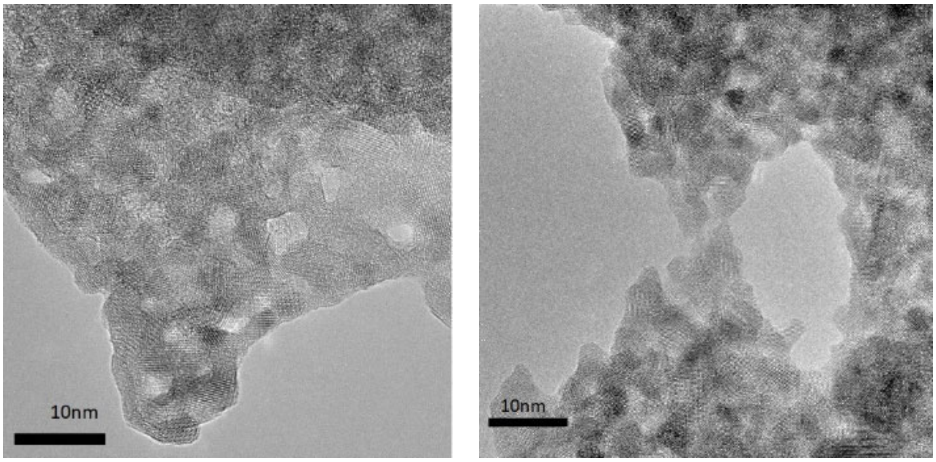

Fig. 6. HR-TEM images of Fe_2O_3 nanoparticles

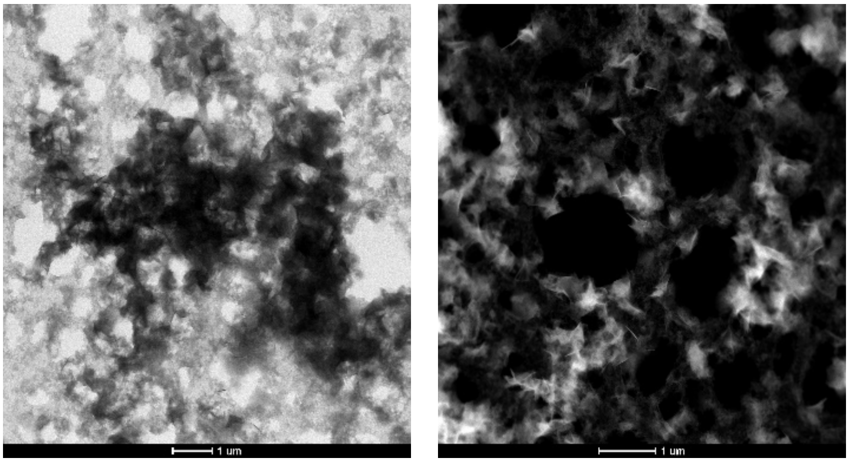

Fig. 7. TEM images of Fe_2O_3 nanoparticles

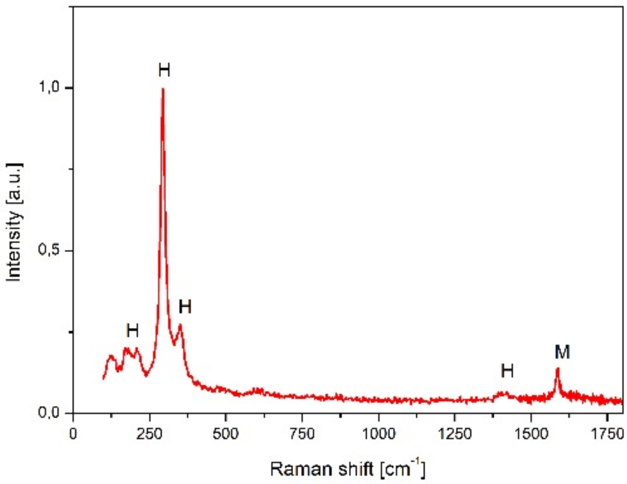

Fig. 8. Raman spectra of Fe_2O_3 nanoparticles (H - hematite, M - maghemite)

Magnetic properties tests using VSM magnetometer (Fig. 9) showed hysteresis loop. In the case of ferromagnetic materials, this graph is characterized by a high coercion and remanence value, which means that there is a clear and wide loop. Paramagnetics show low remanence and no loop in their case. Superparamagnetics show a very narrow loop and a high level of remanence, i.e. their shape resembles the letter S, which in the middle runs like a linear function. High coercion value and wide hysteresis loop informs about magnetically hard materials.

Nanostructured materials are usually magnetically soft and in these cases the loop narrows. The loop obtained as a result of VSM tests widens in the middle part to the shape of the narrow letter S. The remaining curve resembles a linear function. The maximum magnetic induction (remanence) is approx. 1.5 T and the value of the maximum magnetic field is approx. 1.65 kA / m. It indicates that obtained materials exhibit the characteristics of ferromagnetic and super-paramagnetic structure.

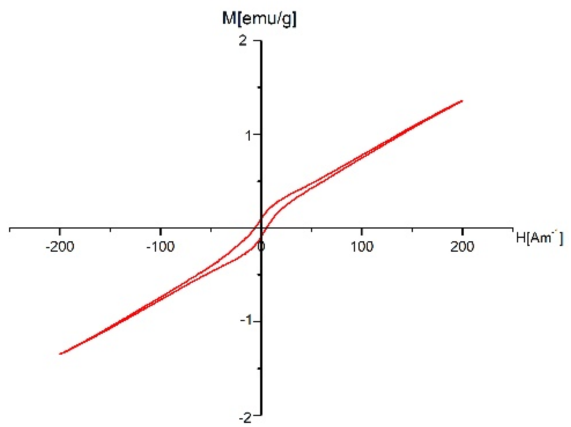

Fig. 9. Hysteresis loop of Fe_2O_3 nanoparticles, received as a result of VSM magnetometer probe

4. Conclusions

As a result of the experiment, iron (III) oxide powders were successfully produced. Morphological studies have shown that the obtained nanoparticles do not exceed 10 nm, which means that they can be considered for use in the phenomenon of hyperthermia. EDS analysis confirmed the chemical composition of the obtained material.

Raman spectra showed the presence of two varieties of iron (III) oxide - hematite and maghemite. Magnetic properties tests proved that powder is mixture of ferromagnetic and superparamagnetic domain, which is characteristic for compound of hematite and maghemite. Those specific factors are the key to possibilities of using the generated particles in cancer therapy.

Acknowledgements

This work was supported by the Ministry of Science and Higher Education of Poland as the statutory financial grant of the Faculty of Mechanical Engineering, Silesian University of Technology in 2020.

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