Multi-Knowledge Electronic Comprehensive Journal For Education And Science Publications (MECSJ)
Issues (48) 2021
ISSN: 2616-9185
www.mecsj.com

Magnetic Iron Oxide Nanoparticles (IONPs) Synthesis and Characterization

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1 Introduction

Nanotechnology is the term used to refer to the manipulation of matter at a molecular, supramolecular and atomic scale. It is the science of controlling materials on a molecular or atomic scale with the aim of developing the microscopic device. Nanotechnology can also be defined as the molecular engineering of functional systems. In the engineering of these systems, nanotechnology utilizes materials referred to as nanoparticles. In nanotechnology, the term nanoparticle is described as a small object which behaves like a small, whole, independent unit in its properties and transportation.

The size of nanoparticles varies from 100nanometer to 1 nanometer[7]. They are usually the size of small molecules and can't be seen with a microscope. Nanoparticles have different properties that they derive from their constituent components. The difference in characteristics and properties of nanoparticles has resulted in them being categorized into different groups based on their properties[5]. One of the most widely reviewed classes of nanoparticles is magnetic nanoparticles. The term magnetic nanoparticle is used to refer to a particular class of nanoparticles that are susceptible to manipulations through magnetic fields. Magnetic nanoparticles are usually made up of two constituent components namely the magnetic material which is usually nickel, cobalt and iron and the chemical component which possesses the nanoparticles functionality.

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The variation in magnetic material leads to the formation of different types of magnetic nanoparticles. There are four main types of magnetic nanoparticles: (1) oxides, (2) ferrites with shell, (3) metallic and, (4) metallic with the shell. Iron oxide nanoparticles, which are also referred to as ferrite nanoparticles, are the most commonly explored form of magnetic nanoparticles in the world. Iron oxide nanoparticles refer to iron oxide particles whose diameters range between 100nanometer and 1 nanometer. There are two main categories of iron oxide nanoparticles, namely magnetite and maghemite which is the oxidized form of magnetite.

Iron oxide nanoparticles have been widely investigated primarily due to their magnetic attributes. To effectively discuss iron oxide nanoparticles, it is essential to review the history of magnetic iron oxide. In-depth research into magnetic iron oxide started in the year 1930 when Louis Neel and William Fuller held a discussion on the magnetic concepts of single domain particles. However, it is important to point out that magnetic iron oxides have been used by humans for centuries.

One of the most widespread uses of magnetic iron oxide in the past was in in-vitro diagnostics as a contrast. Advancements in the synthesis of magnetic iron oxides were driven by fundamental scientific interest in the material and its numerous technological applications. The numerous technological applications of magnetic iron oxide are one of the primary reasons why they are considered to be

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critical. Magnetic iron oxide nanoparticles can be used in magnetic resonance imaging, drug delivery, bioseparation, magnetic hyperthermia and some other activities. Magnetic iron oxides are also regarded to be paramount due to their unique magnetic properties. The unique magnetic properties of magnetic iron oxides enable them to have certain applications.

2 Properties of Magnetic Iron Oxide

1 Bulk Properties

1.1 Structural Properties

According to Teja & Koh (2009), the structure of magnetic iron oxides is associated with a crystal structure that contains an inverse spiral design. The unit cells of magnetic oxides comply with the face-centered cubic pattern that has a crystal lattice parameter[10].

1.2 Physical Properties

Magnetic iron oxide particles exhibit a metallic luster. Hematite is reddish-brown in color, ferrihydrite is yellowish-orange, and magnetite is jet black and opaque. The surface areas of magnetic iron oxides vary by the synthesis method used[2].

1.3 Thermal Properties

Magnetic iron oxides have average melting and boiling points of 1590 degrees Celsius and 2623 degrees Celsius, respectively. The heats of fusion,

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vaporization, and decomposition for magnetic iron oxides stand at 138.16, 298.0 and 605.0 kJ/mol respectively.

1.4 Electrical Properties

Magnetic iron oxides have electrons which when thermally delocalized migrate within the magnetic metal oxide leading to high conductivity exchange constants that range from -28 J·K to 3 J·K between the metal iron oxides' tetrahedral/octahedral and octahedral/octahedral sites.

1.5 Magnetic Properties

Magnetic iron oxides have a Curie temperature of approximately 850K. As the temperature increases to 850K, the metal oxide experiences thermal fluctuations which destroy its ferromagnetic alignments on the tetrahedral sites. This leads to the diminishments of the magnetic iron oxides ferromagnetic strength. At 850K the iron oxides net magnetization turns to zero, and it exhibits superparamagnetic behavior[2].

2 Properties of Nanoscale magnetite

2.1 Structural Properties

As the particle size of a magnetite is decreased towards the nanoscale, there is a corresponding increase its lattice parameter. A decrease in the particle radius of a magnetite will lead to an increase in its Laplace pressure thereby reducing the external pressure that is exerted on the magnetite particle. The lower external pressure leads to swelling of the particle which in turn leads to unit cell expansion.

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The magnetite has a crystal structure which remains constant even with the swelling[2].

2.2 Physical Properties

Magnetite nanoparticles have a surface area of 100 m2g-1. Magnetite colloidal solutions have a jet black color that is also reflected in bulk scale magnetite. It is essential to indicate that magnetite nanoparticles are also assumed to be nonporous.

2.3 Thermal Properties

A decrease in the diameter of magnetite nanoparticles is likely to contribute to lower melting points. The reduction in melting point results in a subsequent reduction in the particles' decomposition, the heat of fusion and vaporization points[2].

2.4 Electrical Properties

Due to the closely packed nature of electrons, magnetite nanoparticles have very few free electrons and thus are regarded as semiconductors of electricity.

2.5 Magnetic Properties

A decrease in the particle size of a magnetite contributes to a reduction in its ferromagnetic properties which in turn enhances the particles superparamagnetic

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behavior. Increasing the temperature of the particles enhances the thermal energy which contributes to magnetic reorientation[1]. As a result of this, magnetite nanoparticles demonstrate remanence in hysteresis loops and zero coercivity[2]. In the presence of an external magnetic field, a magnetites particles induced magnetic field is larger than that of a bulk magnetite[2]. This empowers nanoscale magnetite particles with enhanced capabilities of magnetic separation.

3 Synthesis

3.1 Co-precipitation

Fe_3O_4 Magnetic Nanoparticle Synthesis Procedure.

3.1.1 Materials

The materials utilized in the synthesis of magnetic iron oxide nanoparticles through the co-precipitation procedure are dextran, iron (III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), Iron (II) chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$) and an ammonium hydroxide solution (NH_4OH) in the Nano-Bio-Engg Laboratory, in Southeast Missouri State University.

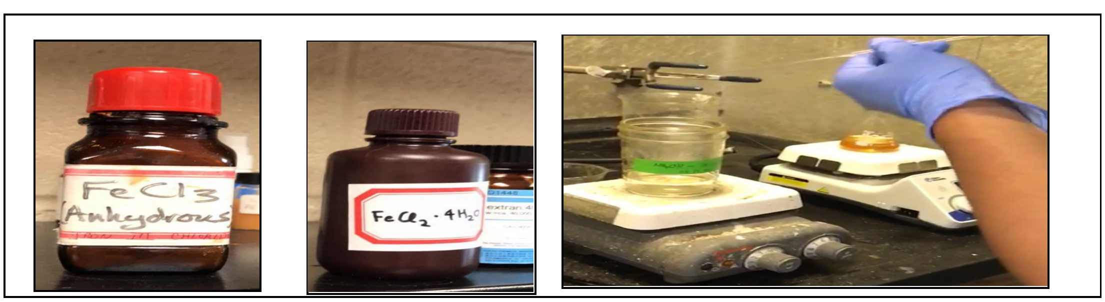

Figure: 1 shown the materials when used in the Nano-Bio-Engg Laboratory

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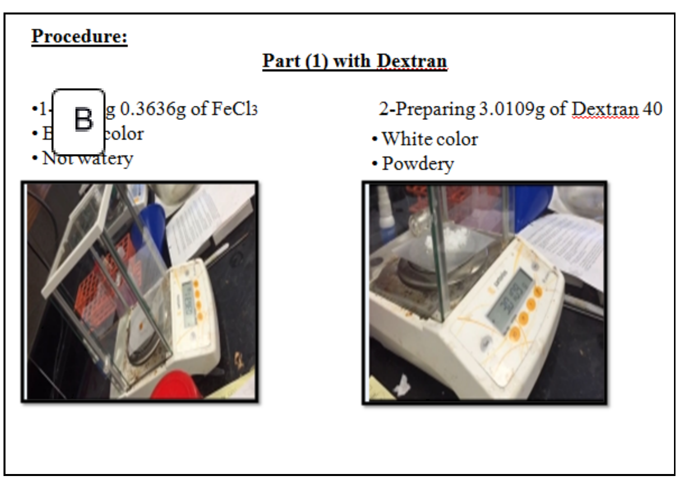
3.1.2 Methods

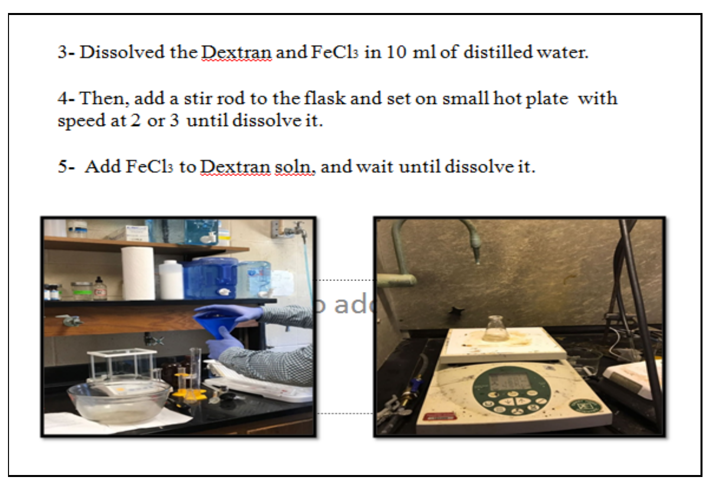
The synthesis was based on the concept of combining the iron (III) chloride hexahydrate with the iron (II) chloride tetrahydrate and the ammonium hydroxide solution to obtain iron oxide, sodium chloride, and water.

$$2FeCl_3 \cdot 7H_2O + FeCl_2 \cdot 4\ H_2O + 8NaOH \longrightarrow Fe_3O_4 + 8\ NaCl + 20\ H_2O$$

The iron oxide would then be heated for 30 minutes at 60 to 75 degrees Celsius to obtain magnetic iron oxide particles.

In performing the synthesis with dextran, the researchers took 0.3636 grams of iron (III) chloride hexahydrate ($FeCl_3$), which is brown in color[12], and dissolved it with 3.0109 grams of Dextran 40, which is white in color and powdery, in 10 ml of purified water in a flask. This was then placed on a hot plate and stirred until the mixture had dissolved. The next step has the addition of 0.1546 grams of $FeCl_2$ into the solution. This was followed by the addition of 25 drops of NH_4OH to the solutions which lead to a change in the color of the mixture, indicating that nanoparticles had started to form.





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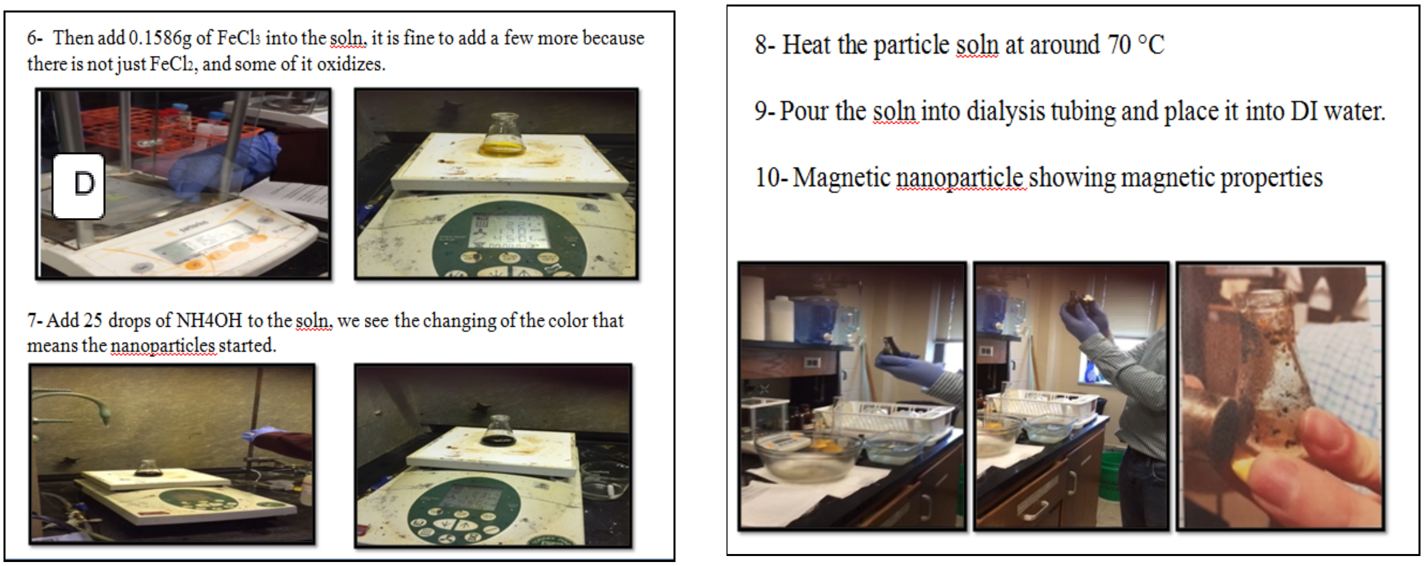

Figure: 2 shown A, B, C, D Procedure when we did in the Nano-Bio-Engg Laboratory

3.1.3 Chemical Precipitation

A temperature of 70 degrees Celsius was applied to the solution to heat it for approximately 30 minutes; before it was poured into a dialysis tube and placed in distilled water. Magnetic nanoparticles that exhibit magnetic properties are formed at this stage. When performing the synthesis without dextran same process was repeated but without adding dextran[6].

3.1.4 Results

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A noticeable difference when using dextran is that the sugar was not properly dissolved with the iron particles. Also, it is essential to not that when dextran was added; the researchers did not observe any magnetic properties[6]. However, when dextran was not added, the iron oxide particles exhibited magnetic properties. The magnetic iron oxide nanoparticles can be verified by using a magnetic bar[6].

3.2 Thermal Decomposition

The co-precipitation method of synthesizing magnetic iron oxide nanoparticles makes it impossible to control size distribution and particle size. Thermal decomposition synthesis of magnetic iron oxide has proven effective in solving the problem that is caused by co-precipitation[3]. Thermal decomposition strategies can be categorized into conventional reaction strategies where the reaction mixture is made at room temperature after which it is heated in an open or closed reaction vessel and the hot-injection approaches which entail the injection of the precursors into a heated reaction mixture.

Because most of the reactions in thermal decomposition of coordinated iron or organometallic precursors contained in organic solvents occur at high temperatures, one obtains narrow size distribution, higher monodisperse and crystalline magnetic iron oxide nanoparticles[12]. Thermal decomposition also provides for the separation of nucleation from growth; one can be able to avoid complex hydrolysis reactions thus producing magnetic iron oxide nanoparticles

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that have superior properties than those obtained through co-precipitation. The most common ferric salts used in thermal decomposition are iron oleate, Prussian blue, ferrocene, and urea complex[11].

In the obtainment of monodisperse iron oxide nanoparticles, one adds various organic molecules such as 1-octadecene, oleic acid, 1-tetradecene and glutamine to the reaction to act as stabilizers[12]. The stabilizers serve to slow down the nucleation process thus affecting the adsorption of nuclei additive and nanocrystals growing[9]. This restricts the growth of iron oxide nanoparticles and in turn favors the formation of tiny iron oxide nanoparticles. The products obtained are usually 30 nm and spherical in shape with a size distribution that can be controlled to a slight extent.

3.3 Hydrothermal Synthesis

A hydrothermal synthesis is a non-aqueous form of synthesizing magnetic iron oxide nanoparticles from various wetchemical methods of crystallizing the intended product in a sealed container from a non-aqueous or aqueous solution at high temperature and under high vapor pressure. This method has proven to be effective in the growing of dislocation-free single particles of crystals. The grains formed through this process also pose a better crystallinity than those formed through the other process of magnetic iron oxide nanoparticles synthesis. Through the use of hydrothermal synthesis, one can obtain high crystalline iron oxide nanoparticles that include Fe_3O_4, γ-Fe_2O_3, and α-Fe_2O_3 nanoparticles[13].

Hydrothermal synthesis provides individuals with the ability to generate crystalline phases that are not stable at their melting point. The synthesis method also provides for the growth of high-quality nanocrystals while at the same time allows for the maintenance of good maintenance of their composition. Hydrothermal synthesis is a conventional and facile method of getting hollow iron oxide nanopar ticles. In a typical procedure, one uses ferric ions (Fe_3^+) as the iron resource. Then urea, acetate, and sodium citrate are mixed in ethylene glycol through stirring[13].

The resultant dispersion is then transferred to an autoclave that is stainless steel and Teflon line and it is sealed to heat at approximately 200 degrees Celsius for between 8 to 24 hours. The route allows for the production of iron oxide nanoparticles that have a controllable shape and size. In conclusion, it is logical to indicate that the hydrothermal method of synthesis allows for the production of magnetic iron oxide that has short nanotubes and other interesting shapes.

4 Characterization

4.1 Size and shape of IONPs

The co-precipitation synthesis of magnetic iron oxide nanoparticles leads to the developments of nanoparticle with small sizes. Through the use of facile

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chemical co-precipitation methods, one can generate IONPs (stands for iron oxide nanoparticles) that have a size of 25 nanometers[12]. The IONPs are nonspherical and stead crystalline.

It is essential to note that in a co-precipitation synthesis, the shape, size, and composition of the IONPs is based on the experimental parameters, such as the type of iron salts used in the synthesis, their ratio, the medium ionic strength and the pH value. Despite its numerous advantageous, a major drawback of this type of IONP synthesis is that it results in quite a broad particle size that is toxic[8].

When using the thermal decomposition method for magnetic iron oxide nanoparticles, the stabilizers act to slow down the nuclei process, and by doing so, affect the growing of the nanocrystals. As a result, the crystals that are obtained through this process are spherical in shape and have sizes below 30 nm[13]. It is important to note that the shape and the size of the particle can also be tailored in such a way that is capable of altering the shape and size of the ions through the additives, solvents, and precursors used. The IONP derived from hydrothermal synthesis are miniature in size and have strong crystalline structures[3].

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4.2 Transmission Electrons Microscopy

Transmission electron microscopy is a microscopy technique where a beam of electrons is passed through the specimen to form an image. The specimen is usually an ultra-thin specimen that is less than 100 nanometers. Transmission electron microscopy is used in the investigation of the characteristics of different IONP's particles as it is capable of viewing tiny particles. Transmission electron microscopes are used for the analysis of IONPs because they provide investigators more insight on the incident that is being reviewed.

Another type of microscope that can aid with the study of IONPs is the scanning electronic microscope which is commonly referred to as SEM. SEM can be defined as an electron microscope that is capable of producing sample images by scanning the surface of the sample with focused electron beams. SEM is capable of achieving a resolution that is better than 1 nanometer, thus making this technique extremely effective.

4.3 Magnetometry

Magnetometry can be described as the technique of mapping and measuring magnetism patterns in the soil. From the discussions, it is evident that the heating of magnetic nanoparticles under AC conditions serves to increase the ferromagnetic standing of the nanoparticles, thus serving to make them more powerful as magnets. In magnetometer, it is essential to point to the fact that in

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some instances the magnetic structure of soils can be altered as a result of the bacteria in the sand.

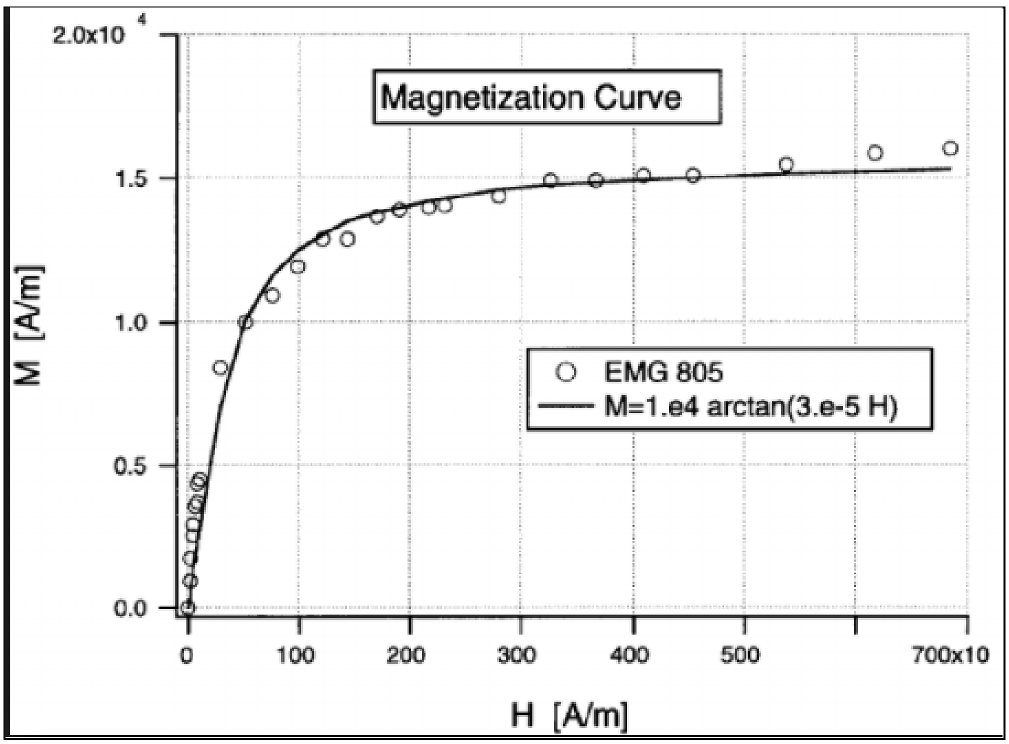

Figure 3: Magnetization Curve

4.3.1 Superparamagnetic nanoparticles

Superparamagnetic nanoparticles possess unique magnetic properties that make them very appealing to researchers. In the use of solution based chemical synthesis, one can obtain control on the size of the nanoparticles and monodisperse the magnetic NPs as a result of the standard deviation in their diameter by 10%.

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4.3.2 Ferromagnetic NP's

Ferromagnetic materials are NP's that have atomic magnetic moments which possess an equal coupling interaction and magnitude between the electrons of the materials thereby serving to increase the magnetic power of the ferromagnet.

5 Conclusion

From the discussion and the articles provided, it is logical to conclude that the constituent components of magnetic iron oxide nanoparticles play a significant role in determining the properties of the matter. Magnetite is the most widely reviewed magnetic iron oxide because of its properties. The magnetic attributes of the magnetite serve to make it one of the most versatile forms of iron oxide nanoparticle in existence since it's the attributes provide it with a number of applications.

It is also evident that the nanoparticles have physical and chemical properties which are built on the synthesis process and the individual components that make up the particles. One also realizes that the synthesis process used has a significant impact on the characteristics of the nanoparticles. Different methods of synthesis use different IONP's development processes, which places great constraints on the nanoparticles[4]. It is also important to note that magnetic iron oxides are unsafe and should be handled with extreme care. This is important because magnetic iron oxides are developed under certain stringent conditions that

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serve to enhance the toxicity of these substances[12]. However, studies in in-vitro fertilization have served to prove that if used the correct way, magnetic iron oxides have minimal impact on the users. According to the study, magnetic iron oxide should be treated effectively to ensure that it is not exposed to any substances that might render it toxic and serve to make it dangerous to the final user who utilizes products made from these components.

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