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SYNTHESIS CHARACTERIZATION AND APPLICATION OF IRON OXIDE NANOPARTICLES IN SURFACTANT ASSISTED MEDIA

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ABSTRACT

Recently, iron oxide nanoparticles (NPs) have garnered a lot of attention due to the exceptional qualities that they possess. These properties include superparamagnetism, surface-to-volume ratio, larger surface area, and an easy separation methodology. For the purpose of synthesising magnetic nanoparticles with appropriate surface chemistry, a number of different physical, chemical, and biological approaches have been utilized. In this study, the methodologies for the synthesis of iron oxide nanoparticles (NPs), the regulation of size and morphology, and the magnetic characteristics of these nanoparticles are summarized, along with contemporary uses in bioengineering, commercial, and industrial settings. There is a significant amount of potential for iron oxides to be utilized in the domains of life sciences, including agriculture, biomedicine, and environmental science. In order to further improve the nontoxic conduct and biocompatible applications of magnetic nanoparticles, a specific surface coating containing organic or inorganic molecules can be applied. These molecules can include surfactants, medicines, proteins, starches, enzymes, antibodies, nucleotides, nonionic detergents, and polyelectrolytes. Hyperthermic treatment of patients can also be accomplished by directing magnetic nanoparticles (NPs) to an organ, tissue, or tumor by the utilization of an external magnetic field. The purpose of this review is to describe latest knowledge on iron nanoparticles, ranging from their synthesis to their characterisation and uses. This study was prepared with the current interest in iron nanoparticles in mind.

KEYWORD: *Iron oxide nanoparticles, Synthesis, Applications.*

INTRODUCTION

Nanotechnology has garnered a significant amount of interest in a variety of study fields over the course of the past twenty years. This interest has been directed towards the creation of nanoscale materials, which can be acquired through a variety of ways, including chemical and physical approaches. Due to the fact that nanoparticles have diameters that range from one to one hundred nanometers, they possess particular and controllable qualities that are distinct from those that they exhibit on the macroscopic scale. This enables them to be utilized in applications that are not found anywhere else. Both of the following are the primary causes of the change in properties: (i) surface effects, also known as the size reduction effect (when the size of a particle is decreased, a greater proportion of atoms are located at the surface); (ii) quantum confinement, which is a modification in the electronic structure.

The ratio of the number of surface atoms to the number of heavy atoms is known to grow dramatically with the reduction in particle size. This is a well-known fact. Nanostructured materials display considerably different physical, chemical, optical, mechanical, electrical, and magnetic properties. This is due to the fact that the surface atoms have less coordination in comparison to the bulk atoms. According to Yetter et al. (2009), the tremendous amount of energy that surface atoms possess is a significant contributor to the amazing properties that nanoparticles possess. As a result, nanoscale materials have a significant potential for applications in a variety of technological fields, including nanoelectronics and computer technology, medicine, aeronautics and space exploration, biotechnology, and agriculture, among others.

In recent years, nanoscale transition of metallic oxides, such as iron oxide, including hematite, magnetite, and maghemite, has been attracting growing interest. This is due to the fact that these oxides exhibit distinctive electrical, optical, and magnetic properties, which can be utilized in a wide variety of applications. These applications include the production of inorganic pigments, magnetic storage media, the development of gas sensors as well as electronic and optical devices, information storage, color imaging, magneto caloric refrigeration, bioprocessing, ferrofluid technology, and wastewater treatment adsorbents.

OBJECTIVES

1. To study iron oxide nanoparticles.
2. To study synthesis.

IRON OXIDES

One of the mineral compounds that can be found in nature in large quantities is iron oxide. Additionally, it possesses a variety of structural and magnetic properties, in addition to exhibiting more than one crystal structure.

Hematite, magnetite, and maghemite are the three of these minerals that are most commonly found. The crystal structure of the three oxides can be described in terms of closely packed planes of oxygen anions that include iron cations at interstitial sites that are either octahedral or tetrahedral.

Magnetite (Fe_3O_4) is one of the most fascinating crystallographic phases of iron oxide, particularly in its nanosized forms. This is due to the fact that magnetite may take on a significant number of different shapes. It also possesses four distinct crystalline polymorphs, each of which possesses its own set of magnetic characteristics. Hematite (α-Fe_2O_3) and maghemite (g-Fe_2O_3) are the two primary forms that can be found in nature. The other oxides, which are in the forms beta (b-Fe_2O_3) and epsilon (e-Fe_2O_3), are nanometric structures that are often created in the laboratory.

Hematite, symbolized as α-Fe_2O_3, is the most well-known of all iron oxides. It is also the most common polymorph that can be found in nature as a mineral, and it may be found in a wide variety of rocks and soils. This oxide exhibits a weak ferromagnetic or antiferromagnetic activity at ambient temperature. It is classified as an oxide. In addition, this material exhibits paramagnetic behavior above 956 K, which is the Curie or Curie temperature, the Néel temperature, or the Curie-Weiss equation. A corundum-type and rhombohedral structure is exhibited by α-Fe_2O_3. This structure is composed of an ion O^{-2} reticle, which is arranged in a close-packed hexagonal crystallographic system, and Fe^{+3} ions, which occupy two-thirds of the octahedral interstices in alternate layers. The synthesis of hematite is simpler than the synthesis of other forms of oxide since it is the final product of the transformation of other forms of iron oxide. Furthermore, hematite is exceptionally stable under environmental circumstances.

In addition to being a typical ferromagnetic mineral, maghemite (g-Fe_2O_3) is thermally unstable and undergoes a transformation into hematite when subjected to higher temperatures. The spinel crystal structure of this material is comparable to that of magnetite, with the exception of the presence of vacancies in the cation sublattice. Two thirds of the sites are filled with Fe (III) ions, and they are placed in a regular pattern, with two sites that are filled being followed by one site that is empty. When subjected to an external magnetic field, g-Fe_2O_3 and magnetite (Fe_3O_4) exhibit a high magnetic response because they are quickly magnetized and so exhibit a high magnetic response. Due to the fact that they are metastable oxides in the oxidative atmosphere, they undergo oxidation to α-Fe_2O_3 when subjected to a temperature that exceeds 673 microkelvin.

Methods for the preparation of iron nps

A variety of procedures, including wet chemical processes, dry processes, and microbiological techniques, are utilized in the preparation of iron oxide magnetic nanoparticles (Figure 1) that possess the required surface chemistry. Iron oxide magnetic nanoparticles (NPs) with adequate surface chemistry can be created using a variety of ways (Figure 1), including wet chemical processes, dry processes, or microbiological approaches. A comprehensive comparison of synthesis methods is included in Table 1, with the intention of assisting researchers, who are currently engaged in this subject, in achieving their goals. Table 1 provides a comprehensive comparison of the various methods of synthesis, with the intention of assisting researchers who are already undertaking work in this area to

1. First, there are the physical approaches, which are complex operations that have the drawback of being unable to control the size of particles in the nanoscale range.
2. Chemical preparation methods: these procedures are straightforward, can be easily implemented, and are effective. They allow for the management of the size, composition, and even the shape of the nanoparticles (NPs). In order to produce iron oxides, it is necessary to add a base to the process of coprecipitation, which involves the combination of Fe^{2+} and Fe^{3+}. The type of salt that is employed, the ratio of Fe^{2+} to Fe^{3+}, the pH, and the ionic strength all play a role in determining the size, shape, and composition of iron nanoparticles that are produced using chemical processes.
3. Techniques based on science.

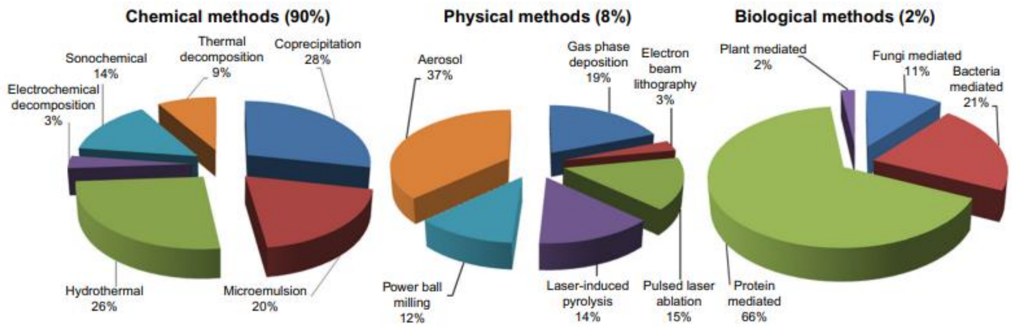

Figure 1 A comparison of the synthesis of SPIONs by three different routes.

Table 1 Iron NP synthesis techniques and their comparison with respect to their product morphology, advantages, and disadvantages

S #	Techniques		Product morphology	Advantages	Disadvantages	References
1	Physical	Deposition of gas phase	Spheres and irregular spheres	Easy to execute	Problematic in controlling the size of particle	22
		Electron beam lithography	Spheres and rods	Well-controlled interparticle spacing	Requires expensive and highly complex machines	30
2	Chemical	Sol–gel method	Spheres, irregular spheres, porous and nonporous spheres, or spindles	Aspect ratio, precisely controlled in size, and internal structure	High permeability, weak bonding, low wear resistance	4
		Oxidation	Irregular elongated and small spheres	Narrow size distribution and uniform size	Ferrite colloids of small size	31, 32
		Chemical coprecipitation	Spheres	Simple and effective	Inappropriate for the synthesis of high untainted, precise stoichiometric phase	24, 28
		Hydrothermal	Elongated, compact irregular spheres, and numerous shapes	Particle size and shapes are easily controllable	High pressure and reaction temperature	23
		Flow injection	Small rods, irregular spheres, sheets, or rhombic shapes	Homogeneity with high mixing with a accurate control of the procedure and good reproducibility	Under a laminar flow regime in a capillary reactor, it requires continuous or segmented mixing of reagents	33
		Electrochemical	Spherical NPs, nanorods, hexagonal nanocrystals, and facets	Controllable particle size	Inability to reproduce	4
		Aerosol/vapor phase	Mesoporous single crystals and small particles, octahedral cages	Large-scale products	Requires very high temperatures	34
		Sonochemical decomposition	Bipyramids, spheres, or truncated rods	Size distribution in narrow particle	Still, mechanism is not well understood	35
		Supercritical fluid method	Mesoporous single crystals, elongated irregular nanotubes	No organic solvents involved and efficient control of the particle size	Requires high temperatures and critical pressure	5
		Using nanoreactors	Spheres, hollow and spherical NPs	Likelihood to specifically control the size of NPs	Complicated conditions	36
3	Biological	Microbial incubation	Small platelets, spherical or rod-like spheres, irregular spheres	Good reproducibility and scalability, high yield, and low cost	Slow and laborious	25

Methods that are based on chemical synthesis are the ones that are most commonly used among these technologies because of their low production costs and high yields. In general, magnetites are produced by adding a base to an aqueous mixture of Fe2+ and Fe3+ chloride at a molar ratio of 1:2, which results in the formation of a black hue.In Equations 1 and 2, the chemical reaction that occurs during the precipitation of Fe3O4 is described. The following is a textual rendition of the overall reaction:

$$Fe^{2+} + 2Fe^{3+} + 8OH \rightarrow Fe_3O_4 + 4H_2O \quad (1)$$

When the atmosphere is devoid of oxygen, it is quite probable that a complete precipitation of Fe3O4 will occur between pH 9 and 14, while the molar ratio of Fe3+ to Fe2+ remains at 2:1. Moreover, Fe3 O4 could be oxidized as follows:

$$Fe_3O_4 + 0{:}25O_2 + 4{:}5H_2O \rightarrow 3Fe(OH)^{3+} \quad (2)$$

It is possible for the conditions to have an effect on the physical and chemical properties of nanoparticles (NPs). In order to prevent iron nanoparticles from oxidizing and aggregating, Fe3O4 nanoparticles are typically coated with molecules that are either organic or inorganic. However, in order to synthesis magnetic nanoparticles, it is necessary to do it in an oxygen-free environment, and it is preferable to do so in the presence of nitrogen gas. Not only does bubbling nitrogen gas prevent oxidation of NP, but it also lowers the size of the particles.

The numerous approaches that were discussed before each come with their own set of benefits and drawbacks (Table 1). Although it is simple to carry out physical procedures, it is challenging to maintain control over the particle size control. The parameters of the wet chemical preparation process allow for some degree of control over the particle size of the substance being prepared. The electrochemical method, the sol–gel method, the supercritical fluid method, the hydrothermal method, the chemical coprecipitation method, the sonochemical decomposition method, the flow injection method, and nanoreactors are all examples of the chemical methods. On the other hand, among all of these methods, the most effective way to get iron magnetic nanoparticles is through the use of aqueous medium. It has been established that variations in the associated parameters, such as the ratio of Fe2+ to Fe3+, the base (NaOH, ammonium hydroxide, and CH3 NH2), and the ionic strength (N(CH3)4+, CH3 NH3+, NH4+, Na+, Li+, and K+), may be used to customize the particle size as well as the polydispersity of the nanoparticles (NPs).4. A number of additional parameters, such as an increase in mixing rate, temperature, the introduction of nitrogen gas, agitation, pH, and the ratio of reactants, are also known to have an effect on the size of the nanoparticles (NPs). However, microbial methods are time-consuming, yet they guarantee low costs, reproducibility, high yields, and scalability. Microbial methods also guarantee high yields.

Characterization of magnetic NPs

Comprehensive surface characterisation approaches, such as surface morphology, chemical composition, and spatial distribution of the functional groups, are utilized in order to achieve a more in-depth comprehension of surface attributes. X-ray diffraction analysis, Fourier transform infrared spectroscopy, transmission electron microscopy, scanning electron microscopy, atomic force microscopy, X-ray photoelectron spectroscopy, vibrating sample magnetometry, and thermal gravimetric analysis are some of the fundamental techniques that are utilized in the investigation of magnetic nanoparticles (NPs). Ion–particle probe, thermodynamic, NP tracking analysis, tilted laser microscopy, zeta-potential measurements, isopycnic centrifugation, hydrophobic interaction chromatography, fieldflow fractionation, electrophoresis, and turbidimetry are some of the other techniques that can be used for characterisation. Table 2 provides a summary of the specific characteristics, levels of success, and any limitations associated with each technique.

Table 2 The analytical techniques for the assessment of the physicochemical properties of NMs

Modalities	Analyzed physical and chemical properties	Successfulness	Restrictions
DLS	Size distribution based on hydrodynamic.	Constructive way for rapid and more consistent measurement. Measures in some liquid media, solvent of interest for monodisperse, hydrodynamic sizes are exactly determined. Moderate expenses on equipment.	With a particular composition, unresponsive correlation of size fractions. Effect of small numbers of large particles in polydisperse sample. Size restrictions. Restricted size determination. Possibility of samples, spherical in shape.
FCS	Dimension, binding kinetics of hydrodynamic.	High temporal and spatial magnification. Uptake sample is low. For studying concentration effect, molecular diffusion, chemical kinetics, and conformation dynamics are specifically performed via fluorescent probes methods.	Due to deficiency of proper methods, it causes limitation in fluorophore species and restriction in usage and inaccuracy.
SERS RS TERS	Size distribution and hydrodynamic size. Conformational variations in structural, chemical, conjugate and electronic characteristics.	No need of sample preparation. Complementary data obtained from IR Capability of detecting tissue abnormality. Improved Raman scattering signal. SERS. Enhanced spatial resolution of the NMs. Topological information.	Compared to Rayleigh scattering, there is comparatively a weak single restricted spatial resolution, enormously minute cross-section. Disturbance of fluorescence irreproducible measurement.
Zeta-potential	Stability concerning to charge on surface	Concurrent measurement of numerous particles.	Electro-osmotic effect deficiency of accurate and repetition measurement.
NSOM	NMs, shapes and size.	Sudden measurement of fluorescence and spectroscopy. Close situation analysis. Nanoscaled surface categorization of chemical information and interactions at nanoscaled declaration.	Lengthy scanning time. Analysis of minute sample area. Intensity of incident light is deficient to stimulate delicate fluorescent molecules. Problems in visualization of soft materials. Analysis restriction toward the NM surface.

CD	In biomolecules. Structural and conformational variations (such as DNA and protein). Thermal constancy.	Constructive and motivated methods.	Conformational fluctuations are due to the involvement of nonspecific residue absorption. Methods are more sensitive than this. For nonchiral. Chromophores, CD signals, are weak. For analysis of molecules consisting of multiple chiral chromophores facing challenges.
MS	Molecular weight. Composition. Structure. Surface properties (secondary ion MS).	High accuracy and precision in measurement. High sensitivity to detection (a very small amount of sample required).	Expensive equipment. Lack of complete databases for the identification of molecular species. Limited application to date in studying NM bioconjugates.
IR ATR-FTIR	Bioconjugate. Surface properties such as structure and conformation.	Rapid and cheap measurement. No or minimal sample preparation demands. Irrespective of sample thickness enhanced reproducibility.	Sample preparation (IR) is complex intervention and efficient absorbance of water. In nanoscale analysis sensitivity is comparatively low.
SEM ESEM	Size and size distribution. Shape. Aggregation. Dispersion.	Simultaneous measurement of the size navigation and shape of NMs. High deliration (below to subnanometer) in natural state visualization of biomolecules supplied by the usage of ESEM technique.	Requirement of conducting sample or coating conductive materials. Need of dry samples. In nonphysiological states, the sample analysis occurs. Size distribution is based on biased statistics. Heterogeneous samples are required. Costly apparatus. For numerous NP bioconjugates, cryogenic method is needed. ESEM resolution is reduced.

Modalities	Analyzed physical and chemical properties	Successfulness	Restrictions
TEM	Shape heterogeneity. Size and size navigation. Dispersion. Accumulation.	With higher spatial resolution than SEM, direct measurement of the size transportation and shape of NMs occurs. For investigation of chemical composition and electronic structure of NMs. A lot of analytical techniques are paired off with TEM.	Ultrathin samples are needed. Requirement of samples in nonphysiological states. Damage or variations in sample. Sampling is insufficient. Equipment is expensive.
STM	Shape heterogeneity. Size and size navigation. Dispersion. Accumulation.	Sudden measurement at atomic level, high, spatial resolution takes place.	Demand of conductive surfaces. Electronic structure and surface topography inevitably having an easy linkage with surface.
AFM	Shape heterogeneity. Size and size navigation. Dispersion. Accumulation and sorption.	Mapping of 3D sample surface resolution of sub-nanoscaled topographic samples. Direct measurement in dry state, ambient, or aqueous environment.	Lateral dimensions over description. Sampling is poor and time consuming The exterior of NM analysis is generally restricted.
NMR	Indirect analysis of size. Structure purity. Concentration. Conformational variations.	Noninvasive and constructive procedure. Minute or less sample preparation required.	Sensitivity is low. Time wasting. Comparatively large amount of sample needed.
XRD	For crystalline materials, shape, size, and structure determination.	Well-organized modalities. At atomic level, high spatial resolution.	Usage in crystalline materials is reduced. Only one binding or conformation site for sample; accessibility compared to electron diffraction is low.
SAXS	Shape, structure, size, and size transportation.	Constructive procedure sample preparation is very simple. Accessibility of amorphous materials and sample in solution.	Resolution is comparatively low.

Applications of iron oxide NPs

Because of their powerful magnetic capabilities, iron oxide nanoparticles were initially utilized in the field of biology, and then later in the field of medicine. These nanoparticles were utilized for the magnetic separation of biological products and cells, as well as for the magnetic guidance of particle systems for site-specific drug delivery. The biodistribution of the nanoparticles is affected by external factors such as the surface chemistry, size, and charge of magnetic particles.Due to the significance that magnetic carriers and particles play in diagnostics and treatment modalities, there has been an increase in the number of activities that have been conducted in clinical applications during the past few decades. A significant amount of interest has been shown in magnetic nanoparticles (NPs) as a labeling material in the biological sciences as well as in a variety of other important areas of the scientific world. A summary of some well-known fields that could potentially benefit from the application of magnetic nanomaterials can be seen in Table 3.

Table 3 The main fields where magnetic NMs have been employed

Fields	Applications of magnetic NPs	References
Biomedical	Magnetic NPs (particularly coated with liposomes) for drug delivery, magnetic hyperthermia, MRI contrast agent, magnetic separation, controlled drug release, cellular therapy, eg, cell labeling, tissue repair, cell separation and handling of cells, purifying cell populations, magnetofection, diseases of the musculoskeletal system, severe inflammation, disability, and pain	9, 31, 99, 113
Health care	Therapeutic targets in chemotherapy (cancer and tumor); nanoscale biosensors and imaging; nanocoatings on surfaces; implants; nanocarrier for vaccination; antimicrobial activities; SLN in drug delivery and research; nanophotothermolysis with pulsed lasers for the treatment of cancer, hepatitis B virus, respiratory syncytial virus, influenza virus, antiviral agents against HIV-1, monkeypox virus, herpes simplex virus type 1, and Tacaribe virus; delivering antigens for a particular disease into the blood stream; preventing aging of the skin	9, 15, 27, 105, 117
Agriculture and food	Nano-based products (nanofertilizers, nanofungicides, nanopesticides), engineered NPs, and CNTs boost crop yields; pyrite NPs are used as a seed treatment for various plants prior to sowing the seeds. Broader leaf morphology, larger leaf numbers, increased biomass. Enhanced breakdown of stored starch. This raises the possibility of developing iron pyrite NPs as a commercial seed treatment agent (pro-fertilizer). The strategy is safe, as the process does not put NPs into the soil. Reduced dose requirement as compared to chemical fertilizers. No adverse effects on plant growth. Nanosensors, nanofood, encapsulation, food packing, nanocoatings, precision farming (remote-sensing devices), nanocomposites, gene transfer (crop improvement), and nanoporous membranes	14, 116, 118, 119
Environmental remediation	Pollution prevention (detection, monitoring, and remediation). Waste water treatment (permeable reactive barriers, membrane filtration, adsorption). Catalyst coatings such as palladium (Pd), climate change (carbon capture), artificial leaf for CO_2 sequestration, mineral carbonation, biomimetic carbonation, N_2O decomposition, methane combustion. Improves manufacturing processes (efficiency, waste reduction), dematerialization (reduction in material quantity), sensing (pollutant sensors, nanoporous membranes, chemical and bio-nanosensors, nanowire sensor for explosives), and energy (heat distribution, eg, ceramic-like materials that provide sufficient reliability and durability of the entire structure)	15, 73, 111, 120, 121
Energy	Photovoltaic film coatings, improved efficiency of fuel production and consumption, fuel cells and batteries, nanobioengineering of enzymes, thermoelectric materials, and prototype solar panels, batteries, aerogels, conversion of waste heat in computers, automobiles, homes, power plants, etc, to usable electrical power	4, 122, 123
Defense and aerospace	Nanocomposites, nanocoatings, sensors and electronics, fuel additives and energy devices, and smart materials	124–126
Construction	Nanocoatings, nanocomposites, nanoscale sensors, smart materials, and additives to concrete. Iron oxide pigments are used in coloring concrete, brick, tile, and other construction materials	127, 128
Automotive	Additives in catalysts and lubricants, nanocoatings, fuel cells, composite fillers, and smart materials	129, 130
Textiles	Sensors, nanofibers, coatings, and smart materials	131, 132
Electronics	Printed electronics, carbon nanotubes, nanoscale memory, nanowires, NEMS, spintronics, and quantum dots	124, 133, 134

CONCLUSION

With the purpose of contributing to research in the field of composite solid propellants, the present article provides a concise presentation of the features of nanosized metallic iron oxides, as well as their methods of synthesis, primary techniques of characterisation, and their application in thermal decomposition of AP. Iron oxides, hematite (α-Fe2 O3), and maghemite (γ-Fe2 O3) are examples of transition metal oxides that have been extensively utilized in catalytic processes and thermal degradation of ammonium perchlorate. These oxides have been classified as nanoscale transition metal oxides. The size of their particles and the surface area of their surfaces have a significant impact on the catalytic activities that they exhibit. Because of the quantum effect, which is caused by the reduction in size, and the surface effect, they are more effective than conventional catalysts. Both of these factors are related to one another. The reduction in particle size leads to an increase in the number of atoms that are placed on the surface, which in turn leads to an increase in the catalytic activity of nanoparticles that contain iron oxide, hematite, and maghemite.

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