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Synthesis magnetic nanomaterials by chemical synthesis route

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Abstract

The chemical manufacture and investigation a substantial amount of interest about physicochemical properties of magnetic oxide microscopic particles due to of the diverse range of fields that may benefit from them, including electronics, biomedicine, and environmental remediation. Chemical approaches in this research, magnetic oxide nanoparticles, particularly Sol-gel synthesis and co-precipitation are two processes that are used to manufacture iron oxide (Fe_3O_4 and $-\text{Fe}_2\text{O}_3$). The main goal is to create nanoparticles that can be regulated regarding dimensions, form, and style, as well as magnetic characteristics while ensuring high chemical stability.

The yielded nanoparticles' characteristics (physical, chemical, and mechanical), such as dimensions, form, and surface charge, crystallinity, and magnetic behavior, are characterized using techniques like many forms of electron microscopy, including three different types of imaging techniques: techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and vibrating sample magnetometry (VSM). The process of surface functionalization and how reaction variables factors impact the level of final nanoparticle's behaviour are also investigated.

The synthesized magnetic oxide nanoparticles exhibit superparamagnetic behavior, with their size and magnetic properties tunable by adjusting synthesis conditions. This makes them suitable for various applications, including administration of drugs, MRI scans, and organic reactions. The results of this research add to our knowledge of the correlation between synthesis techniques and the resulting magnetic and chemical properties, offering insights for tailoring nanoparticles for specific technological and industrial applications.

Keywords: Synthesis magnetic, nanomaterials, chemical, superparamagnetic, SEM

Introduction

Among the most significant areas of study in contemporary science is nanoscience. Researchers in the fields of science, engineering, chemistry, and healthcare are enabling breakthroughs those working in healthcare and the life sciences by using nanotechnology, which is enabling them for use in cellular and molecular processes. The distinct size and physicochemical characteristics of nanoparticle [NP] materials make them very useful. Since they may be used for so many different things, several researchers have concentrated on finding ways to create different kinds concerning biotechnology, healthcare, engineering, and ecological preservation using magnetic nanoparticles (MNPs).

Currently, there are few practical applications of nanostructured materials in the field of biology. The stellar qualities of these materials, however, bode well for their

potential applications in the near future. Particles as small as a nanometer exist in the space between molecules and structures as small as a micron. These particles are known as nanoclusters. Because of their microscopic size, these materials display properties rarely seen in bigger structures (not even 100 nm). On the other hand, their enormous size makes them accessible to hitherto inaccessible domains of quantum behavior when seen as molecules. Numerous recent breakthroughs science, biology, and chemistry have all experienced developments on this magnitude. Nanocrystal properties are strongly influenced by their dimensions, making the fabrication of mono-disperse-sized nanocrystals crucial. In research of the physicochemical characteristics of nanocrystals that vary with their size, it is crucial to synthesize monodisperse-sized nanocrystals with controlled sizes.

Magnetic recording media, medicinal compounds for cancer

therapy, MRI contrast agents, and are only only a few of the many applications for magnetic nanoparticles in industry. Harnessing the power of magnetic nanoparticles' characteristics is crucial for each potential application. When used for data storage, for instance, particles must be able to maintain a consistent, programmable magnetic state to stand in for data bits that are immune to changes in temperature. At ambient temperature, particles that display superparamagnetic behavior include recommended for use in biological applications.

Additionally, under physiological circumstances, the stability of the magnetic particles is crucial, and this includes water with a pH of 7, for use in therapeutic, biological, and diagnostic applications. For this fluid to be colloidally stable, the particle size-that need to be diminutive enough to avoid being deposited by gravity, as well as the surface charge-which produces steric and coulombic counter repulsions. For use in live or cultured organisms in biomedical research, more particle limitations might be used. In order to keep the magnetic nanoparticles from changing their original structure, clumping together, or biodegrading when exposed to a biological system, they must be enclosed in a biocompatible polymer either during or after preparation for *in vivo* applications. Entrapment, adsorption, or covalent attachment are all mechanisms by which the polymer-coated nanoparticles may bind medicines. Magnetite, iron, nickel, and cobalt are examples of magnetically sensitive components; the size, core, and coatings of the particles, as well as their ultimate dimensions, are the primary determinants of biocompatibility and toxicity in these materials. Most often nanoparticles with iron oxide groups for use in medicine include magnetite (Fe₃O₄) or g-Fe₂O₃, the oxidized version of magnetite.

Nanoparticle

Typically, inorganic materials are meant when the word "nanoparticle" is used, rather than individual molecules. Ever since Granqvist and Buhrman, two pioneers in the field, performed the first thorough foundational research in the US and Japan, respectively, utilising nanoparticles-then called ultrafine particles-in the 1970s and 1980s, the terms are typically used interchangeably. On the other hand, the US National Nanotechnology Initiative didn't start until the 1990s, when the word "nanoparticle" was already widespread. When compared to tiny particles or bulk materials, nanoparticles may display size-related characteristics that are vastly different. Nanoparticles have a lengthy history, despite their association with contemporary science. Using nanoparticles, craftspeople in Mesopotamia in the 9th century would give pots a shimmering appearance, while Romans used them in the famous The cup that Lycurgus held in the fourth century composed of dichroic glass. There is typically a noticeable metallic shimmer of gold or copper on Renaissance and Middle Ages pottery.

Glazing with a metallic layer applied on its clear surface gives off this sheen. If the film has managed to withstand weathering processes such air oxidation, its shine could still be evident. The film's sheen is due to the uniform dispersion embedded in the vitreous ceramic glaze are nanoparticles of copper and silver. The craftspeople made these nanoparticles by mixing vinegar, ochre, clay, salts and

oxides of copper and silver, then applying it to earthenware that has been glazed in the past. After that, it was fired in a kiln with an atmosphere that is reduced to a temperature of around 600 °C. It was possible for migrate to the outer layers of the glazing, where copper and silver ions are as it became more malleable due to the heat. The color and visual effects are provided by metal nanoparticles that were created when the ion was reduced back to metal in the reducing environment.

To put it simply, nanoparticles connect large-scale materials to their atomic or molecular constituents, which is why they pique scientists' curiosity. The physical characteristics of a bulk material are size-independent, but at the nanoscale, these qualities become apparent. Nanoparticles' impressive and unexpected properties are because, primarily, of their enormous surface area, which more than makes up for their little mass. The use of nanoparticles in solar cells is one example; by the manipulation Unlike thin films of continuous sheets of material, particles may be engineered to absorb more sunlight depending on their size, shape, and composition. allows for control of solar absorption. Due to their microscopic size, titanium dioxide nanoparticles are invisible, but they have a self-cleaning action. One of the reasons zinc oxide nanoparticles are often utilized in sunscreen lotion production is that they have better UV blocking qualities than their bulk equivalent. Another reason is because they are photostable. polymers with a greater T_g, along with other enhanced mechanical qualities are the result of adding clay nanoparticles to polymer matrices, which boost reinforcing and make the polymers stronger.

Literature Review

Kai Zhu *et al.* by 2018 ^[1] extraordinary features of magnetic nanomaterials (MNMs), such as super-paramagnetism the effect of heat energy on a ferromagnetic nanoparticle have garnered significant attention throughout the last several decades. Nanoparticle moments are subject to thermal energy fluctuations in the superparamagnetic size domain. A variety of methods for getting ready have been explored in order to provide MNMs with the appropriate structures and characteristics, in an effort to comprehend the basic behavior of super-Para magnetism and to create applicable possible applications. Nevertheless, several challenges must yet be surmounted before we can prepare well defined magnetic nanostructures, such as exchange-coupled nanomagnets, the most sophisticated magnets to come.

Alla Divya Naga Aparna *et al.* (2022) ^[2] Researchers are very interested on the possible use of nanoparticles derived from iron oxide in many different areas. The biodegradability, adaptability, biocompatibility, and unique magnetic characteristics of magnetite have prompted much research into this substance. To ensure high-quality nanoparticles for their intended applications, precise control over the manufacturing process is required, because surface chemistry, dimensions, and form of nano-scale magnetite have a direct impact on its behaviour. There have been a number of physical, chemical, and biological processes employ in published works and by various enterprises and laboratories.

Margarita L. Alvarado-Noguez *et al.* (2023) ^[3] A coating of polyphenolic groups (-OH and -COOH) on the surface was provided to magnetite (Fe₃O₄) nanoparticles ~12 nm in this

study by means of creating and applying a coating on *Curcuma longa* L. extract. Various bio-applications are set in motion by this, which also aids in the creation of nanocarriers. A propensity for being coupled to iron ions is found in the plant extracts of *Curcuma longa* L., a ginger (Zingiberaceae) relative. Iron oxide superparamagnetic nanoparticles (SPIONs) were determined by analysing their low remanence energy, coercive field (H_c) of 26.67 Oe, and tight hysteresis loop (M_s) of 8.81 emu/g. In addition, the produced nanoparticles (G-M@T) exhibited addressable cores at 90-180° and controllable anisotropy in a single magnetic domain and connects with that domain in one direction. Surface analysis was used to identify the distinct sulfur, iron, and carbon peaks.

Siti Kamilah Che Soh *et al.* (2018) [4] In order to create palladium (II) complex supports, utilising a co-precipitation technique, magnetite nanoparticles (MNPs) of varying colours were produced. These MNPs may one day serve as a robust solid foundation for homogenous systems. In an alkaline medium, $FeCl_3 \cdot 6H_2O$ and $FeCl_2 \cdot 4H_2O$ were mixed to complete the two-hour synthesis. The technique was carried out in an inert environment to improve iron oxide nanoparticle properties. The magnetic resonance sampler (MSM), the- physicochemical characteristics of this support were characterized by the use of FESEM, TGA, XRD, and FTIR spectroscopy, to name a few.

Synthesis of nanoparticles

A three-dimensional object with at least two dimensions between 1 and 100 nanometres is considered a nanostructure, and the synthesis process may produce many nanostructures. The synthesis of nanoparticles may be accomplished in several ways. Using these methods, one may create both dry particles and nanoparticles dispersed in liquid. Building a nanostructure from the atomic level down to the nanoparticle level is one such approach.

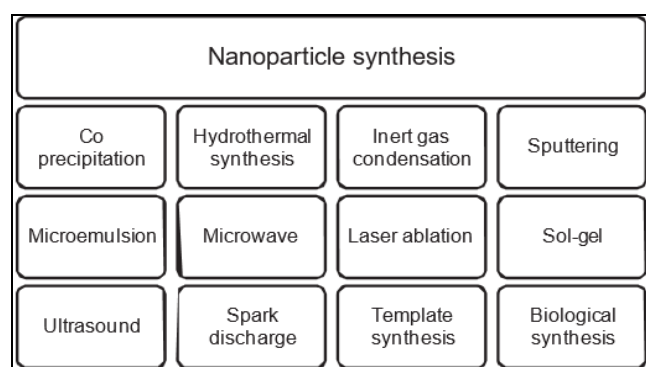


Fig 1: Methods for creating nanoparticles

Coprecipitation

Responding to coprecipitation, nucleation, growth, coarsening, and agglomeration all happen at the same time. These are the features that coprecipitation processes display:

- Byproducts typically consist of insoluble species that form at very high supersaturation levels.
- There will be a ton of little particles created during the crucial process of nucleation.
- Ostwald ripening and aggregation are two examples of secondary processes that significantly alter product size, shape, and characteristics.

Hydrothermal Technique

Many various types of scientists and technicians have taken an interest in the hydrothermal method, which has been widely praised. The word hydrothermal has its origins only in the field of geology. Roderick Murchison (1792–1871) of Britain was the first to use the term to characterize the process by which water under high pressure and temperature altered the mantle, a process that produces a variety of minerals and rocks.

There has been success with hydrothermal synthesis in producing several valuable solids, including microporous crystals, chemical sensing oxides, ECS, magnetic materials, luminescence phosphors, complex oxide ceramics and fluorides, and superionic conductors. Additional distinctive condensed materials that may be created by this approach include gels, thin films, nanoscale particles, stacking-sequence materials in particular, and distinguishing helical and chiral structures.

Hydrothermal synthesis is the process of creating new chemicals by reacting existing ones in a controlled environment at temperatures and pressures higher than room temperature and pressure.

To create single crystals by hydrothermal means, minerals must be soluble in very hot water at very high pressure. The autoclave is a steel pressure vessel that is used to produce crystals. It is supplied with water and nutrients. To facilitate nutrient dissolution at one end of the growth chamber and seed development at the other, the chamber is designed to keep two opposite ends at a constant temperature differential.

Hydrothermal and solvothermal synthesis has many benefits

It is possible to make almost any substance soluble in the right solvent by bringing the system up to a pressure and temperature close to its breaking point.

Hydrothermal/solvothermal synthesis has many advantages over solid-state synthesis, including the potential to replace it and the ability to produce materials that would otherwise be inaccessible. It also significantly improves chemical reaction between the two substances.

Magnetic Nanoparticles

The functional parts of magnetic nanoparticle conveyors include a magnetic core, a surface coating, and an operationalized outside coating. The particle may be magnetically controlled by an externally applied field thanks to the superparamagnetic core located within the carrier. The application determines the process of magnetic center creation. As an example, the two most recognized nontoxic minerals for therapeutic purposes are minerals like magnetite (Fe_3O_4) and maghemite (γFe_2O_3), for example, which possess high oxidative reliability. Although magnetic centers made of neodymium-ironboron, nickel, and cobalt might improve magnetic characteristics, these materials could be detrimental to human health or easily oxidized (Vadala M.L. *et al.* 2005) [8]. Possible ways for generating magnetic nanoparticles include physical vapor deposition (Willard M.A. *et al.* 2004) [9], mechanical attrition (Bonnemann H *et al.* 2004) [10], and chemical approaches (Inouye K *et al.* 1982) [11].

Magnetic nanoparticles may avoid attraction, repulsion, and

the possibility of magnetic aggregation because of their small size, which allows them to operate independently of gravity and magnetic field direction. (Rosenzweig R. E *et al.* 1985) [12]. Then, the attractive forces of van der Waals could draw particles together. A surface coating that provides steric repulsion (references to the atomic system in the molecule) may be required to inhibit such particle-framework communication, ensure solidity, and prevent aggregation.

One of the most significant obstacles in almost every application is the synthesis technique of nano-materials, which dictates the particle's magnetic properties, surface chemistry, size, shape, and distribution of sizes. Material particles with ferritic and ferromagnetic properties, including Fe₃O₄ and some alloys, take on an uneven shape when milled from their bulk forms. But they may become spherical when made from gas phases and aerosol, by wet chemistry, plasma atomization, or another process. Additionally, crystalline or amorphous particles may be created in a solution that is shaped like a sphere., depending on the formation process. The former is when the crystallites are disordered, while the latter is when they are organized. Furthermore, the synthesis procedure greatly influences the magnetic behavior of the particle by deciding the amount and distribution of structural flaws or impurities. There have been a lot of recent efforts to find ways to make "monodispersed colloids" including uniformly sized and shaped nanoparticles. The physicochemical characteristics of each individual particle are a perfect reflection of the system's overall uniformity in these systems. For both basic research and modeling purposes, monodispersed colloids have been used to evaluate properties that are size and shape dependent quantitatively. Furthermore, it is now obvious that well-defined powders with established qualities are the best starting point for achieving the quality and repeatability of commercial goods. Powders like this have found use in many fields, including medicine, ceramics, photography, printing inks, and catalysis.

Superparamagnetic, loops that are displaced upon field cooling, fields with high saturation, irreversibility of the field, and extra contributions from anisotropy are among the unique and noteworthy phenomena shown by magnetic nanoparticles. The magnetic the majority of the influence on the behavior of individual nanoparticles comes from both surface effects and those of limited size, which give rise to these phenomena. It was first predicted by Frenkel and Dorfman that ferromagnetic material particles less small than a certain threshold, usually below 15 nm would have a single magnetic domain, meaning they would be uniformly magnetized regardless of the applied field. When these particles are heated once the blocking temperature is crossed, their magnetization behaviour may be described as superparamagnetic, the same as atomic Para magnetism, with the caveat that very high susceptibilities and, by extension, moments, are involved.

Main Synthesis Methods of MNPs

Innovative biomedical uses, including such as MRI, tailored medication delivery, and healthcare diagnosis and treatment, have been made possible by the creation of biocompatible, functionalized ferromagnetic nanoparticles. Using magnetic particles that are either embedded in or spread in a polymer

matrix is essential for achieving biocompatibility.

Nanoparticles made of magnetic polymers have been synthesized as well as derived from natural sources. The magnetic carriers' fraction of magnetic material is reduced and their particle diameter is increased by this method. Maintaining tight precise regulation of magnetic nanoparticle size is of paramount importance. Ideal particles for use in biomedicine should be nanoparticles ranging in size from 5 to 20 nanometres. Making surface modifications to magnetic particles with small molecules offers an alternative to magnetic polymer nanoparticles and opens the door to the prospect of complementary attachment to functional biomolecules as well as sol stability. Two critical phases are necessary in order to successfully synthesize the altered magnetic particles: first, modifying the magnetic particles' surfaces chemically; and second, synthesizing magnetite nanoparticles with size control.

The chemical and physical characteristics are significantly impacted by the manufacturing procedure of magnetite nanocrystals. It is for this reason that the literature reports a variety of approaches. Second, modifying surfaces during the production of functionalized magnetic nanoparticles requires magnetic particles is chemically modified. Amino acids have been the subject of several investigations as chelating agents for maghemite (γ -Fe₂O₃) and magnetite (Fe₃O₄), a procedure that offers dual benefits in water treatment applications. In addition, the amino acids themselves are completely harmless. Using these two methods, the modified magnetic particles are synthesized in all of these investigations.

There has been a lot of research over the past decade on different ways to synthesize MNPs. Various synthetic methods are employed to generate MNPs that fulfill the defined requirements for size, shape, stability, and biocompatibility. A few of typically used techniques for making MNPs Chemical pulverization, chemical precipitation, microemulsion, sol-gel, and biological procedures. Showing the physical, chemical, and biological processes that go into making MNPs.

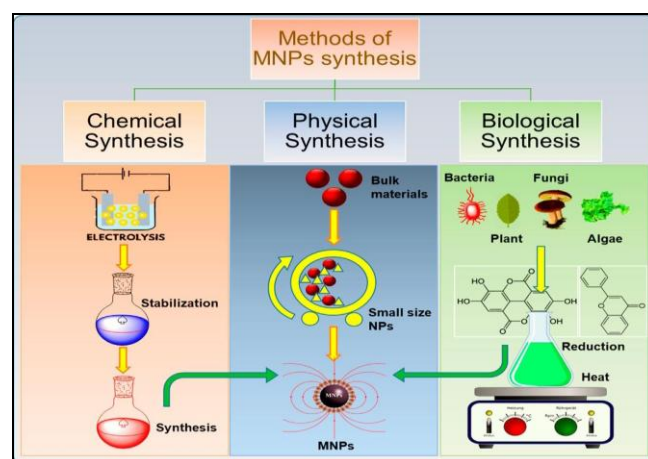


Fig 2: General methods of MNPs synthesis.

Physical Methods

There are physical procedures that use both "top-down" and "bottom-up" approaches. The top-down approach comprises cutting down of bulk materials to nanoparticles through high-energy ball milling processes. It is quite difficult to

mechanically crush nanoparticles to the required size and form.

Method of Ball Milling/Mechanical Method

Milling raw materials using a ball mill is one top-down way to make MNPs. A straightforward and effective approach is mechanically crushing particles with coarse textures into particles with small textures. In 1970, this technique was first introduced. A compact, cylindrical, hollow container with a number of steel balls within is used to grind the raw materials; this is the basic operating concept. The process produces nano- or micro-sized powder by constantly crashing steel balls in contact with solids, hence transferring motion to the material. Some of the most important the milling time, several parameters influence the process of nano/micro size crystal formation, including ball size, vibration speed, and ball-to-powder ratio. The primary problem with the method is that it might contaminate the product. In contrast to particles made by chemical synthesis, these ones have a broad size range

Conclusion

Nanoparticles of transition metal ferrite exhibit magnetic characteristics that are size dependent, much like bulk materials. What size is the particles do not substantially affect the formation of spinels. The magnetic characterization aligns with data collected by XRD, SEM, TEM, and PL investigations. The process of creating NiFe₂O₄ nanoparticles is simple, quick, and inexpensive, which could significantly expand their potential applications. The essential of this material is tested for the oxidation reactions. The findings suggest a possible redistribution for the essential elements involved in the as synthesized Nickel ferrites with subsequent unique properties of this NiFe₂O₄. This approach is posited to serve as a superior method for the preparation of NiFe₂O₄ nanomaterials.

This work presents a method characterized by numerous advantages, including exceptional flexibility, consistent quality reproducibility, cost-effective production, and ease of handling. The ability to regulate particle size with exceptional purity leads to the attainment of favorable characteristics on the scale of nanostructures. Finally, a approach that allows for the effective synthesis of high-crystalline ZnFe₂O₄ straightforward, economical microwave combustion technique. The investigation revealed shown that the fuels utilized greatly affect the foundational, morphological, optical, and magnetic characteristics.

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