

Magnetic Iron Oxide Nanoparticle Synthesis

Subjects: **Others**

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Iron oxides are chemical compounds which have different polymorphic forms, including γ -Fe₂O₃ (maghemite), Fe₃O₄ (magnetite), and FeO (wustite). Among them, the most studied are γ -Fe₂O₃ and Fe₃O₄, as they possess extraordinary properties at the nanoscale (such as superparamagnetism, high specific surface area, biocompatible etc.), because at this size scale, the quantum effects affect matter behavior and optical, electrical and magnetic properties. Therefore, in the nanoscale, these materials become ideal for surface functionalization and modification in various applications such as separation techniques, magnetic sorting (cells and other biomolecules etc.), drug delivery, cancer hyperthermia, sensing etc., and also for increased surface area-to-volume ratio, which allows for excellent dispersibility in the solution form. The current methods used are partially and passively mixed reactants, and, thus, every reaction has a different proportion of all factors which causes further difficulties in reproducibility. Direct active and complete mixing and automated approaches could be solutions to this size- and shape-controlled synthesis, playing a key role in its exploitation for scientific or technological purposes. An ideal synthesis method should be able to allow reliable adjustment of parameters and control over the following: fluctuation in temperature; pH, stirring rate; particle distribution; size control; concentration; and control over nanoparticle shape and composition i.e., crystallinity, purity, and rapid screening. Iron oxide nanoparticle (IONP)-based available clinical applications are RNA/DNA extraction and detection of infectious bacteria and viruses. Such technologies are important at POC (point of care) diagnosis. IONPs can play a key role in these perspectives. Although there are various methods for synthesis of IONPs, one of the most crucial goals is to control size and properties with high reproducibility to accomplish successful applications. Using multiple characterization techniques to identify and confirm the oxide phase of iron can provide better characterization capability. It is very important to understand the in-depth IONP formation mechanism, enabling better control over parameters and overall reaction and, by extension, properties of IONPs. This work provides an in-depth overview of different properties, synthesis methods, and mechanisms of iron oxide nanoparticles (IONPs) formation, and the diverse range of their applications. Different characterization factors and strategies to confirm phase purity in the IONP synthesis field are reviewed. First, properties of IONPs and various synthesis routes with their merits and demerits are described. We also describe different synthesis strategies and formation mechanisms for IONPs such as for: wustite (FeO), hematite (α -Fe₂O₃), maghemite (γ -Fe₂O₃) and magnetite (Fe₃O₄). We also describe characterization of these nanoparticles and various applications in detail. In conclusion, we present a detailed overview on the properties, size-controlled synthesis, formation mechanisms and applications of IONPs.

iron oxide nanoparticles (IONPs)

formation mechanisms

reproducible

biomedical

1. Introduction

Iron oxide nanoparticle (IONP)-based technologies are catalyzing rapid developments in nanotechnology. Due to technological importance, extensive research has been carried out on the development of various synthetic routes to yield IONPs with desired properties^[1]. Among IONPs, mainly Fe_3O_4 and $\gamma\text{-Fe}_2\text{O}_3$ are extensively studied^[2]. In general, iron oxides are classified into different phases (magnetite, hematite, maghemite, wustite). In the nano form, a material possesses interesting optical, magnetic, and electrical properties which cannot be found in their bulk form. This phenomenon can be described as the "quantum size effect"^{[3][4][5]}. In the nanometer range of IONPs, the quantum effect dominates the behavior-affecting magnetic, electric, and optical properties of the matter. In the nanoscale, there is an impact of specific individual atoms or molecules, while in the bulk form, property is attributed to the average of all the quantum forces that affect all of the atoms. For example, magnetic Fe_3O_4 nanoparticles are superparamagnetic below the size of 20 nm^[6]. As the nanoparticle size decreases, this property tends towards paramagnetic or superparamagnetic magnetization. Therefore, a decrease in nanoparticle size will enhance superparamagnetic behavior and decrease ferromagnetic behavior. As the size of nanoparticles decreases, the relative oxygen concentration decreases; therefore, a slight reduction in the iron valance state occurs. Because of this ferrous ion content increase, an increase in magnetization should also be observed^[7]. Similarly, $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles have gained technological importance due to their magnetic and catalytic properties. High magnetization and hysteretic heating make them potential candidates in separation and biomedical areas, and the semiconducting property and chemically active surface allow catalytic activities such as photocatalytic ability^{[8][9]}. Iron oxide nanoparticles (IONPs) have a broad range of significant applications in electronics^{[10][11]}, biomedicine^{[12][13][14]}, energy^{[15][16]}, agriculture^{[11][18]}, and animal biotechnology^{[19][20]}, as shown in Figure 1. In a small size of about 10–20 nm, the superparamagnetic properties of Fe_3O_4 and $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles become apparent, therefore, better performance can be achieved for the above-mentioned applications. Additionally, due to the increased surface-to-volume ratio, they show excellent dispersability in solutions^[21].

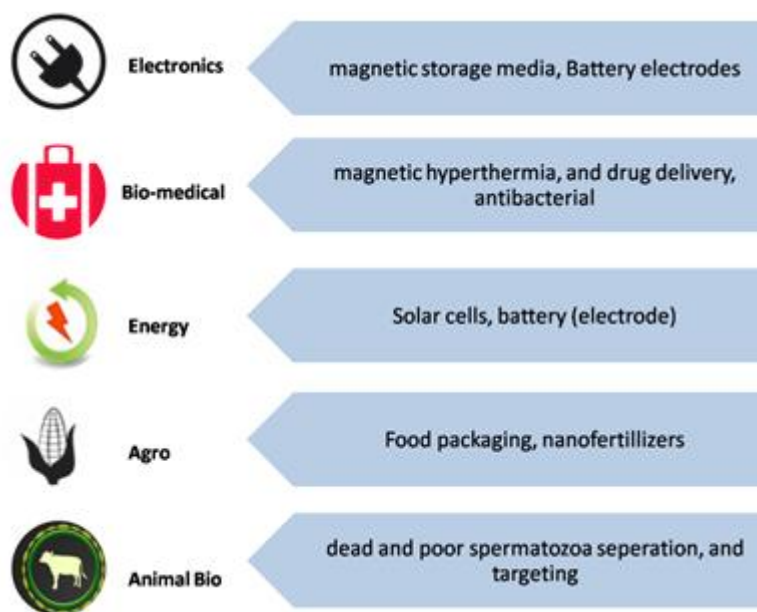


Figure 1. Various applications of iron oxide nanoparticles (IONPs).

However, reproducible synthesis of IONPs with desired properties is still a problem^[22]. This is because existing synthesis methods show a passive approach towards synthesis reaction. The main challenges and key points to overcome them are explained in Figure 2. In existing methods, reactants are mixed partially and passively. Unreacted components therefore effect the final product when undesired reactions takes place, as the proportion of all these factors is different in every reaction, making it difficult to achieve reproducibility in the desired properties^[23]. Immediate purification of nanoparticles after reaction becomes necessary to minimize error. Direct active and complete mixing of reactants and automated approaches could solve this issue. Researchers are mainly focused on size- and shape-controlled synthesis, as size determines the surface area, which plays a key role in its exploitation for scientific or technological purposes^[24].



Figure 2. Challenges and key points in reproducible synthesis of nanoparticles.

2. Manipulation of reaction parameters

Manipulation of reaction parameters is necessary to obtain controlled nanoparticles in terms of size, shape, purity, crystallinity, and morphology. A synthetic route should enable control over reaction parameters: temperature; concentration; fluctuation in temperature; pH; stirring rate; particle distribution; size control; control over shape; nanoparticle composition and structure, which includes crystallinity, purity, rapid screening, and reliable adjustment of parameters^{[22][25][26][27]}.

In our opinion, the established synthetic routes of iron oxide nanoparticles have difficulty in controlling the particle size, shape, and properties. Many of the reported methods have their own pros and cons, as described in Table 1. It is necessary to develop a new synthetic route for IONPs that yields nanoparticles in a reproducible manner with excellent size control. This review explains various dimensions associated with synthesis of IONPs and their applications, and different synthesis mechanisms are summarized. Figures S1–7 represented in supplementary

materials corresponds to various IONPs synthesis methods graphically presented which also includes key points for each corresponding method.

Table 1. Merits and demerits of different IONP nanoparticle synthesis methods.

Type of Synthesis	Pros	Cons	Reference
Microwave	Short reaction time, higher yields, excellent reproducibility, easy handling	Expensive, unsuitable for scale-up and reaction monitoring	[28][29]
Spray pyrolysis	Finely dispersed particles of predictable size, shape and variable composition	Aggregated particles, expensive	[30][31][32]
Laser pyrolysis	Small particle size, narrow particle size distribution, near absence of aggregation	Complicated, very expensive	[21][31]
Pulsed wire discharge method	Fast process, higher purity of NPs	Batch process, limited production, high vacuum systems, costly process, contaminations in product	[33][34]
Chemical vapor condensation	Suitable for preparing small quantities to demonstrate desired properties in the laboratory	Low production, difficult to control size and particle size distribution	[35]

Co-precipitation	Convenient method, simple and rapid preparative method, easy control of particle size and composition	Extensive agglomeration, poor morphology and particle size distribution	[36][37][38]
Thermal decomposition	Producing highly monodispersed particles with a narrow size distribution	High cost, long-time synthesis reaction, high temperature	[39][40][41]
Microemulsion	Monodispersed nanoparticles with various morphology can be produced	Not very efficient and difficult to scale up	[39][42]
Polyol	Uniform size particles can be prepared, easy to scaleup	Needs high temperature, long time	[22][31]
Sol–Gel	Low processing cost, energy efficiency, high production rate, and rapid productivity	Limited efficiency, high cost	[43][44][45]
Sonochemical	Simple, low cost, safe, environment friendly, absence of many reactants	Very small concentration of prepared NPs, particle agglomeration is very narrow	[33][46]
Biological synthesis of nanoparticles	Selectivity and precision for nanoparticle	Limited knowledge, difficulty in controlling	[47][48]

using plants and formation, cost effective, size and properties
bacteria eco friendly

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