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Chapter

New Approaches in Synthesis and Characterization Methods of Iron Oxide Nanoparticles

Cristina Chircov and Bogdan Stefan Vasile

Abstract

Recent years have witnessed an extensive application of iron oxide nanoparticles within a wide variety of fields, including drug delivery, hyperthermia, biosensing, theranostics, and cell and molecular separation. Consequently, synthesis and characterization methods have continuously evolved to provide the possibility for controlling the physico-chemical and biological properties of the nanoparticles to better suit the envisaged applications. In this manner, this chapter aims to provide an extensive overview of the most recent progress made within the processes of iron oxide nanoparticle synthesis and characterization. Thus, the chapter will focus on novel and advanced approaches reported in the literature for obtaining standardized nanoparticles with controllable properties and effects. Specifically, it will emphasize the most recent progress made within the microwave-assisted, microfluidics, and green synthesis methods, as they have shown higher capacities of controlling the outcome nanoparticle properties.

Keywords: iron oxide nanoparticles, synthesis processes, green synthesis processes, characterization techniques, physico-chemical properties

1. Introduction

Iron oxides are transition metal oxides ubiquitously found in nature, having many implications in various biological and geological processes [1–4]. They occur naturally as aggregates, mineral nanoparticles, or nanostructured coatings onto other soil grains [5], being an essential biogeochemically-active component of the Earth ecosystem [6]. Moreover, iron oxides are formed in a variety of polymorphs with different stoichiometric and crystalline structures [2, 3, 7], including oxides, e.g., wüstite or ferrous oxide (FeO), magnetite (Fe_3O_4 or $FeO{\cdot}Fe_2O_3$), maghemite (γ-Fe_2O_3), ε-Fe_2O_3, hematite (α-Fe2O3), and β-Fe_2O_3, hydroxides, e.g., iron(III) hydroxide (bernalite) and iron(II) hydroxide, and oxyhydroxydes, e.g., goethite, feroxyhyte, akaganeite, and lepidocrocite [2, 3, 7–13]. Among them, magnetite, maghemite, and hematite are crystalline and most commonly used in biomedical and pharmaceutical applications, while other forms, such as goethite, are amorphous and occur at high pressure and temperature conditions [2, 3, 7, 14]. Furthermore, iron oxides can also be categorized based on their electrical properties into insulative, i.e., ferrous oxide, conductive, i.e., magnetite, and semiconductive, i.e., hematite and goethite [1].

By contrast, iron oxides can be processed into nanoparticles with magnetic properties, which further allow for their manipulation by external magnetic fields [7, 15]. Among them, magnetite nanoparticles are, by far, the most intensively studied, as they have demonstrated considerable potential in a myriad of applications, including drug delivery, magnetofection, hyperthermia, photoablation therapy, magnetic resonance imaging as contrast enhancement agents, theranostics, biosensing, bioanalysis through biological labeling, tracking, and detection, bioseparation, antimicrobial therapies, tissue engineering and regeneration, wound healing, catalysis, nanorobots, ferrofluids, microelectronics and ultrahigh density magnetic storage media, magnetic paints, pollutant removal sorbents, and batteries [7, 16–22]. Additionally, recent studies have shown an intrinsic peroxidase activity of iron oxide nanoparticles, which could be further exploited in applications such as biocatalysis, wastewater treatment, detection tools, magnetic enzyme-linked immunosorbent assay kits, or artificial enzymes [23–29].

Evidently, each of the previously mentioned applications require specific nanoparticle properties [30]. In this context, it has been confirmed that the physico-chemical properties of magnetite nanoparticles, namely, size, shape, stability, crystal structure and crystallinity, chemical composition, and surface area, energy, and roughness, significantly determine their magnetic properties and, consequently, their biological behavior, drug concentration, toxicity, and efficacy [19, 31–33]. Moreover, studies have shown that the synthesis route of magnetite nanoparticles greatly impacts their physico-chemical properties, thus highlighting the necessity to improve synthesis performance by enhancing standardization, automation, monitoring, and mass production [17, 19, 33].

Presently, the most ubiquitous synthesis method for magnetite nanoparticles is the co-precipitation of ferrous and ferric ions through the addition of an alkaline solution [34]. Although it is a simple and cost-efficient method characterized by considerably high productivity [32], its reproducibility is still limited due to the presence of the intermediate phases within the final product. Additionally, it does not allow for the precise control of nanoparticle size and shape, which further leads to significant variations in the physico-chemical properties of the final product [32, 34–36]. Therefore, there is a fundamental need for the exploration of novel synthesis processes that could further ensure optimal, controllable, and scalable properties. In this context, iron oxide nanoparticles obtained from natural, green sources are continuously gaining the interest of the scientific community as they provide a potential alternative to overcome the limitations of conventional nanoparticles [37]. Consequently, characterization techniques should also be advanced to ensure a reliable assessment of nanoparticle properties. In this manner, the variety of magnetic nanoparticle applications, ranging from biomedicine and pharmaceutical industry to data storage, could greatly benefit from such improvements.

Therefore, this chapter aims to provide an updated overview of the most recent developments within the field of magnetite nanoparticle synthesis and processing, as well as the most advanced characterization techniques utilized for evaluating their properties and potential.

2. Novel iron oxide nanoparticles synthesis methods

There are two well-established approaches involved in the synthesis of nanoparticles, namely, top-down and bottom-up approaches. Generally, top-down methods involve the crushing, breaking, or fractioning of bulk materials into smaller parts to produce

nanoparticles through mechanical action [38, 39]. Such methods include mechanical crushing, milling, or grinding, laser ablation, sputtering, etching, or electron beam deposition, offering an alternative eco-friendly route despite the required high time and power consumption [40–42]. By contrast, bottom-up approaches are based on chemical reactions among specific atoms, ions, or molecules necessary for the formation of nanoparticles. Considering these principles, synthesis routes can be further divided based on the nature of the involved process into physical, that can be associated to the top-down methods, chemical (e.g., co-precipitation, sol-gel, thermal decomposition, emulsion and microemulsion, hydrothermal, and microwave-assisted methods), and biological (which utilize plants or microorganisms for the generation of nanoparticles), the latter two being attributed to bottom-up approaches (**Figure 1**) [38, 39, 43, 44].

Figure 2 depicts a comparison between the most commonly used methods for the synthesis of magnetite nanoparticles. It can be observed that chemical methods comprise the majority of the investigated routes, the co-precipitation method accounting for the highest percentage. Specifically, the co-precipitation process involves two possible pathways, either partial oxidation of iron(II) salts or the aging of a stoichiometric mixture of iron(III) and iron(II) salts through the addition of an alkaline solution that leads to nucleation and growth mechanisms and finally to the generation of Fe_3O_4 nanoparticles. The chemical reaction principle involved in the production of magnetite nanoparticles is shown in Eq. (1):

$$Fe^{2+} + 2Fe^{3+} + 8OH^- \leftrightarrow Fe(OH)_2 + 2Fe(OH)_3 \rightarrow Fe_3O_4 + 4H_2O. \quad (1)$$

Although it is the easiest to implement, time-efficient, and safe method, involving limited use of harmful solvents, the co-precipitation process is considerably disadvantageous in terms of reproducibility and possibility to control the outcome properties of the obtained nanoparticles [46, 47].

Thus, there is a fundamental need for the investigation of novel synthesis routes that could improve the features of magnetite nanoparticles. In this context, recent years have witnessed a shift toward the implementation of previously non-conventional methods that could potentially provide a plethora of alternatives in terms of modulating physico-chemical properties. Thus, the following sections will describe the most recent advancements within the production of magnetite nanoparticles through microwave-assisted, microfluidic, and green synthesis methods.

2.1 Microwave-assisted method

Owing to its numerous advantages, microwave-assisted synthesis has become a particularly attractive method for various synthetic chemistry reactions. Specifically, this technique has provided the means for the easy production of nanoparticles in a considerably time- and cost-efficient manner, with reduced energy consumption and increased environmental friendliness [48–51] through the use of 50% less power than electric furnaces with similar capacities [52]. Besides the associated economic aspects, the microwave-assisted method has received increased scientific interest due to the possibility of tuning the parameters to obtain the desired size and shape of magnetite nanoparticles with significantly narrow distributions and high reproducibility, phase purity, and yield [49–51]. This is possible due to the characteristic uniform heating and nucleation, rapid kinetics and crystallization, and phase selectivity [50, 51].

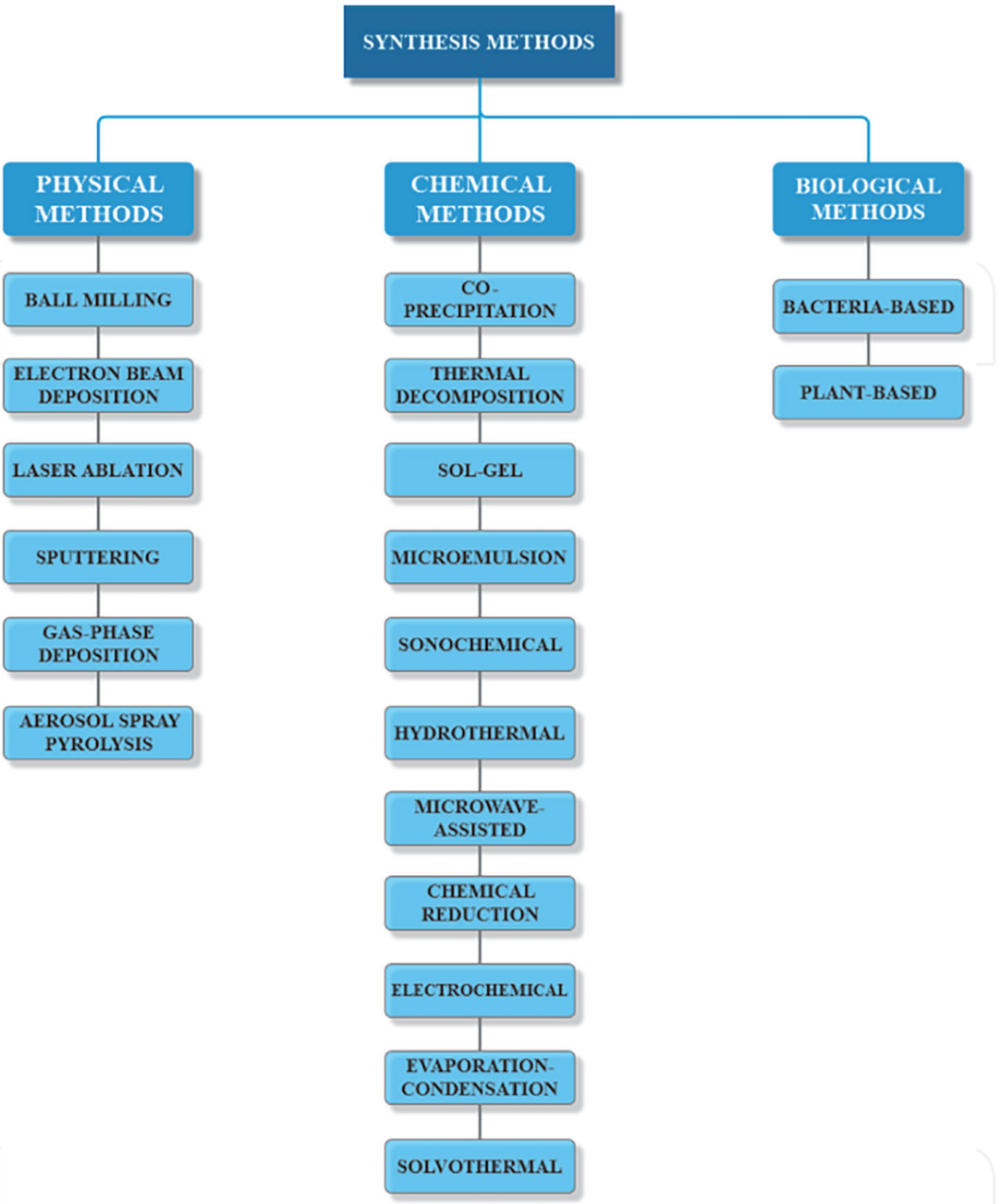

Figure 1.
The main types of magnetite nanoparticle synthesis methods. Reprinted from an open-access source [43].

The basic principle involved in this method is based on the activation and subsequent alignment of dipoles (i.e., mechanism of dipolar polarization) and/or ions (i.e., mechanism of ionic conduction) present within a material through the interactions with microwave electromagnetic radiations. Consequently, internal heating will occur in a highly homogenous manner, thus, leading to a rapid temperature rise that is responsible for reducing the reaction time and the necessary energy [48–52]. In the case of magnetite nanoparticles, it has been demonstrated that the microwave-assisted method offers the possibility to control their magnetic properties by adjusting the experimental parameters [49].

Generally, the microwave-assisted method is combined with other synthesis processes, such as co-precipitation. Thus, the synthesis involves the co-precipitation

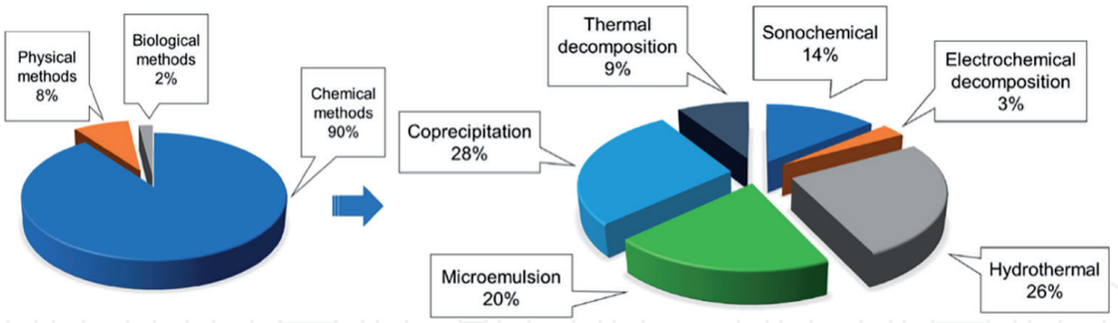

Figure 2.
The prevalence of the most commonly utilized magnetite nanoparticle synthesis methods. Reprinted from an open-access source [45].

of iron oxide nanoparticles through the classical method, followed by the microwave treatment that enables the control over the properties of the nanoparticles. Several studies have demonstrated the possibility to obtain monodisperse iron oxide nanoparticles with well-controlled sizes and high crystallinity, saturation magnetizations, and stability, as compared to the co-precipitation counterparts [53–56]. Additionally, the possibility of developing uniform polyethylene glycol [53], humate polyanion [54], and silica [55] coatings was also demonstrated.

2.2 Microfluidic approaches

Microfluidics is a relatively new field that has brought together fluid dynamics, chemistry, and material science principles for allowing the precise and accurate manipulation of small fluid volumes within microchannels [57, 58]. In this context, microfluidic technology-based methods for the synthesis of nanomaterials have emerged as an alternative to conventional routes that could provide possible solutions to the currently existing limitations. Specifically, microfluidic devices represent synthesis platforms with outstanding features for the fabrication of nanoparticles, including small capillary dimensions and consequent large surface/volume ratios and reduced reagent volume use, rapid and uniform mass and heat transfer, ease of automation, reduced residence time, and precise control of mixing [38, 57–59]. In this manner, by increasing the control of the implicated reaction parameters (e.g., device geometry, flow rate, reagent concentration, reaction time, temperature) [59, 60], nanoparticles with superior uniformity, stability, and encapsulation efficiency and narrow particle size distributions can be obtained in a highly reproducible and controllable manner [38, 57–60].

Based on their geometry, microfluidic devices can be classified into tubular reactors, which generally involve circular channels that are either in-house produced or purchased (most common commercially available reactors have T and Y type junctions), and chip reactors, which involve more complex geometries and are usually in-house manufactured using various fabrication techniques [38]. The working principle of microfluidic approaches for the synthesis of nanoparticles resides on the movement of fluids within microchannels and microchambers with unique geometries to integrate the preparation, reaction, and separation steps. In this context, there are two main types of microfluidic reactors that involve different synthesis strategies, namely, single-phase or continuous-flow microfluidics and multi-phase or droplet-based microfluidics, which can be further divided according to the carrier fluid into gas–liquid and liquid–liquid segmented flows [38, 59].

Microfluidics is currently evolving as a promising alternative for the synthesis of magnetite nanoparticles with controlled size, shape, and surface chemistry that can be modulated according to the application requirements [57, 58]. Since it involves a relatively simple reaction, it can be obtained through both types of synthesis strategies. Continuous-flow microreactors that contain one inlet for the iron precursor solution and one inlet for the alkaline solution will ensure the formation of the nanoparticles at the interface between the two fluid layers if the pH value is high enough for nucleation. While this is usually the preferred route owing to its increased homogeneity and versatility, some applications require faster interactions. Therefore, the multiphase microfluidics involving cross-flow designs are receiving increasing attention. In this approach, the channels containing the precursor solutions, i.e., the dispersed phase, will intersect the channels containing the alkaline solution, i.e., the continuous phase, where the nanoparticles will form and be further transported within the continuous phase [38, 60]. Furthermore, the microfluidic platforms used for the synthesis of magnetite nanoparticles can be made of various materials, such as glass, metals, silicon, or polymers, that must be resistant to the fluids introduced within the microchannels [38].

Although the number of studies is still limited, the results are promising, thus paving the way toward the future of nanoparticle synthesis. In this context, the synthesis of magnetite nanoparticles was investigated through the use of a single-flow polydimethylsiloxane microfluidic reactor [61] and a T-junction polymethylmethacrylate microchip fabricated by a laser cutting machine [57]. Furthermore, another study fabricated magnetite nanoparticles using a 3D flow microfluidic device focused by two basic sheath streams, that were subjected to a postsynthesis surface functionalization step outside the microreactor [62]. Moreover, other studies demonstrated the possibility of developing *in situ* chitosan-coated magnetite nanoparticles using two types of microchip configurations fabricated through 3D printing [63] and by the soft lithography process [64].

2.3 Green synthesis methods

The merge between nanotechnology and biology has led to the rise of a new and highly advanced field of nanomaterial synthesis using living microorganisms of both prokaryotic and eukaryotic origins, such as algae, bacteria, fungi, yeasts, viruses, and plants [65]. Within this framework, the synthesis of nanoparticles via green technologies utilizing microorganisms and plant extracts is continuously emerging as a safe, cost-efficient, renewable, and environmentally friendly alternative [65–68] which does not implicate complex protocols [69] or the use of intermediary base groups [70]. Additionally, green synthesis methods lead to the formation of nanoparticles with higher stability as they do not involve the use of chemicals that increase particle reactivity, enhanced biocompatibility, non-toxicity, and antimicrobial and anticancer properties [66–69]. Such methods are possible due to the resistance mechanisms developed by microorganisms and plants to endure the highly toxic environments generated by high metal concentrations. Specifically, the intrinsic chemical processes of these living entities can remodel inorganic metal ions into nanoparticles to reduce or eliminate the toxic effects. There are two main processes involved in the biogenic synthesis of nanoparticles, namely, through bioreduction, i.e., the reduction of metal ions by intrinsic biological processes, and biosorption, involving the assimilation of metal ions within the cell wall and the consequent formation of stable nanoparticulate structures through the assembly with the present macromolecules [65].

Generally, plant-based synthesis of nanoparticles is more advantageous in terms of higher kinetics, increased reduction and stabilization yield, and easier large-scale production [66, 68, 70]. The plant-mediated formation of nanoparticles can occur intracellularly or inside the plant, through the presence of specific biomolecules (e.g., aldehydes, ketones, flavones, phenols, amino acids, proteins, polysaccharides, tannins, terpenoids, saponins, vitamins), extracellularly, using plant extracts, or through individual phytochemicals. The mechanism involves the linkage between the atmospheric or phytochemical-generated oxygen that reduces the metal ions, followed by the electrostatic interactions between the newly formed metal oxides that will lead to the formation of the nanoparticles. The nature of the phytochemicals is responsible for their size, shape, stability, and reactivity variations [67, 68, 71]. Based on their produced phytochemicals, various plant parts have been investigated, including root, leaf, flower, petal, fruit, stem, peel, or seed [70].

Although green synthesis methods have been mostly applied for obtaining silver, gold, and copper nanoparticles, the synthesis of iron oxide nanoparticles through the use of plants or microorganisms has become an intensively studied field owing to the biocompatible, non-toxic, and stable nature of the final products [72]. Thus, there are many protocols available in the literature for the green synthesis of iron oxide nanoparticles, which generally follow a similar methodology. Briefly, the procedure begins with the starting material preparation and extraction, by collecting, washing, drying, weighing, grinding into a fine powder, boiling in water or methanol/ethanol under continuous stirring, centrifugation, and filtration. Subsequently, the extract is mixed with the precursor salt solutions, such as $FeSO_4{\cdot}7H_2O$, $FeCl_3{\cdot}6H_2O$, $(FeNO_3)_3{\cdot}9H_2O$, $FeSO_4$, $FeCl_3$, $FeCl_2{\cdot}4H_2O$, $FeCl_2$, or $FeSO_4{\cdot}5H_2O$, of varying molarities. Finally, the mixture is heated and vigorously stirred until the color of the solution changes and intensifies according to the type of iron salts utilized. The obtained iron oxide nanoparticle pellets are further washed and dried [73].

The available literature studies reported the synthesis of iron oxide nanoparticles using Bauhinia tomentosa [74], pomegranate seeds [75], Hibiscus rosa-sinensis [76], Mimosa pudica root [77], Carica papaya leaf extract [78], Cymbopogon citratus [79], Ficus carica leaf extract [80], and Platanus orientalis leaf extract [81].

3. Advanced iron oxide nanoparticles characterization techniques

As previously emphasized, the physico-chemical properties of iron oxide nanoparticles often dictate their applications [82]. Since the characterization of nanoparticles is significantly challenging due to the increased interdisciplinarity of the field, it is fundamentally important to characterize nanoparticles to the maximum extent to ensure a more rapid implementation in commercial applications [83]. Generally, the physicochemical properties of iron oxide nanoparticles are evaluated through a variety of different techniques, depending on the parameter that must be determined [35, 73, 82]. **Table 1** depicts the most important characteristics of iron oxide nanoparticles and the suitable characterization techniques for determining them.

The most important parameter to be evaluated is the size and consequently the size distribution of the nanoparticles, as it can affect other properties and determine the behavior of the final product within the envisaged application [35, 83]. Although size measurements within the macroscale might appear trivial, size determinations within the nanoregime might lead to different interpretations depending on the

Nanoparticle property	Characterization techniques
Size	TEM, XRD, DLS, NTA, HRTEM, SAXS, SEM, AFM
Size distribution	DLS, NTA, SAXS
Shape	TEM, HRTEM, SEM, AFM, STEM
Crystal structure	XRD, HRTEM, SAED, STEM
Elemental/chemical composition	XRD, SEM-EDX, ICP-MS, XPS, EELS
Surface area/specific surface area	BET analysis, NMR
Surface charge	Zeta potential
Magnetic properties	VSM, SQUID, Mössbauer spectroscopy, MFM

TEM—transmission electron microscopy, XRD—X-ray diffraction, DLS—dynamic light scattering, NTA—nanoparticle tracking analysis, HRTEM—high-resolution TEM, SAXS—small-angle X-ray scattering, SEM—scanning electron microscopy, AFM—atomic force microscopy, STEM—scanning transmission electron microscope, SAED—selected area electron diffraction, EDX—energy-dispersive X-ray spectroscopy, ICP-MS—inductively coupled plasma mass spectrometry, XPS—X-ray photoelectron spectroscopy, EELS—electron energy loss spectroscopy, BET—Brunauer–Emmett–Teller, NMR—nuclear magnetic resonance, VSM—vibrating sample magnetometry, SQUID—superconducting quantum interference device, MFM—magnetic force microscopy.

Table 1.
The characteristics of iron oxide nanoparticles and the associated characterization techniques. Adapted from an open-access source [35, 73, 83].

characterization technique employed. When referring to nanoparticles, size can be correlated to the atomic structure-defined physical dimension, the diffusion/sedimentation-dependent effective size of the nanoparticle within a matrix or solvent, or the effective size weighted by the mass/electron distribution [84, 85]. Furthermore, size distribution represents an estimation of the quality of the synthesis process, as the general aim is to obtain close to monodisperse nanoparticles [82–84].

The shape also plays a fundamental role upon the behavior of iron oxide nanoparticles, as it can further lead to toxic effects due to cell harming. Therefore, the employed synthesis routes must allow for the control of nanoparticle shape as a crucial parameter [86, 87]. Commonly, electron microscopy techniques are utilized for the precise evaluation of the morphology and consequently the shape of the nanoparticles [83, 88].

Moreover, crystal structure and chemical composition also represent essential characterization steps in the process of iron oxide nanoparticle development [83]. Although X-ray diffraction represents the most common technique for crystal structure, crystallinity, and phases evaluation [89], studies have shown that in the case of nanoparticles with sizes below 5 nm, the diffractogram patterns are influenced [90]. Thus, other, more reliable methods should be developed. Selected area electron diffraction represents an alternative that better depicts the crystal structure of nanoparticles [84]. Chemical and elemental composition determination provides an estimation of the purity of the nanoparticles. Additionally, the chemical composition is a key parameter that can influence the electrochemical activity of the nanoparticles [91]. Moreover, another interesting characterization possibility involves the precise distinction between iron(II) and iron(III) to differentiate the iron oxide phases present within the nanoparticles, which could be possible through the electron energy loss spectroscopy method [92].

Although it might result in high agglomeration degrees, high surface areas of iron oxide nanoparticles are essential for ensuring the desired application [83, 87]. For

example, in waste or pollutant removal applications, iron oxide nanoparticles must possess high surface areas to increase capture and immobilization efficiency [93]. Surface area is determined through straightforward gas sorption techniques, such as the BET analysis [84].

The surface charge can be correlated with the colloidal stability and interactions of the nanoparticles. Specifically, the interactions of iron oxide nanoparticles within the biological fluids will determine the formation of the protein corona on their surface and, thus, the probability of cellular uptake [84]. Generally, the surface charge of nanoparticles is measured through a zeta potentiometer by applying a voltage to the samples. The results are given in terms of zeta potential ζ, which refers to the difference in the electric potential between the particle surrounding stationary charge layers and the potential of the solution [94–97]. Zeta potential values higher than +15 mV and lower than −15 mV are usually attributed to colloidally stable suspensions, as they generate electrostatic repulsions that are strong enough to counteract aggregation of the nanoparticles [82, 84, 85]. However, there are several parameters that could influence the zeta potential, such as the pH and ionic strength of the solvent or the presence of charged/uncharged molecules that can be adsorbed onto the surface of the nanoparticles [84].

Considering the extensive studies performed toward hyperthermia applications for controlled drug delivery and cancer therapy, an essential characteristic of iron oxide nanoparticles is their magnetic behavior. The magnetic properties of iron oxide nanoparticles directly depend upon the synthesis route and the size and shape of the obtained nanostructures [35]. Similar to other properties, the magnetic behavior of nanostructured materials is significantly different than those of the bulk materials, since the size decrease leads to changes from the multidomain to the single domain and, finally, to the superparamagnetic state [83]. Specifically, nanoparticles with superparamagnetic properties are characterized by negligible remanent magnetization and coercive field. Thus, when the external magnetic field is removed, the nanoparticles exhibit no magnetism. By contrast, ferro- and ferrimagnetic nanoparticles feature a magnetic hysteresis, associated with a remanent magnetization, thus requiring a coercive field for reverting the magnetization to zero [35, 98]. Furthermore, the magnetic behavior can also be influenced by the agglomeration and aggregation processes, which further favor dipole-dipole or exchange interactions [99]. There are several methods that can be utilized for the analysis of iron oxide nanoparticle magnetism, each associated with specific sensitivities [35].

4. Conclusions

Iron oxide nanoparticles have been intensively studied for a variety of applications within numerous fields, ranging from medicine and pharmaceutics to microelectronics and analytical chemistry. Since their utilization is continuously rising, the need for improving the currently available synthesis methods is fundamental. In this context, novel preparation routes must be explored to develop uniform and standardized iron oxide nanoparticles. Among the recently implemented strategies, microwave-assisted, microfluidics, and green synthesis methods have demonstrated an undoubted potential toward reaching this goal. Specifically, nanoparticles obtained through these methods were characterized by superior properties as compared to the co-precipitation counterparts. Furthermore, the advancements within the characterization techniques could further lead to new insights and potential improvements

within this area. In this context, the most important techniques often include size, shape, structure, crystallinity, and magnetic behavior determinations. Therefore, the intensive research work investigating the synthesis of iron oxide nanoparticles must further continue.

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Conflict of interest

The authors declare no conflict of interest.

Notes/thanks/other declarations

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Author details

Cristina Chircov[1,2] and Bogdan Stefan Vasile[1,2]*

1 Department of Science and Engineering of Oxide Materials and Nanomaterials, University Politehnica of Bucharest, Bucharest, Romania

2 National Research Center for Micro and Nanomaterials, University Politehnica of Bucharest, Bucharest, Romania

*Address all correspondence to: bogdan.vasile@upb.ro

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