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Review Article

Process of Synthesis and Analysis of Nanoparticles Recovered by Magnetic Methods

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Abstract

Magnetic materials, particularly ferrites, are integral to various electronic and biomedical applications due to their unique magnetic and electrical properties. Ferrites, which typically adopt spinel structures, are synthesized by mixing iron oxide (Fe_2O_3) with other metallic elements, such as nickel, zinc, or manganese. They exhibit ferromagnetic behavior below the Curie temperature and paramagnetic properties above it. Iron oxide nanoparticles (NPs), particularly Fe_3O_4 and $\gamma\text{-}Fe_2O_3$, have gained significant attention for their versatility in fields like catalysis, data storage, and biomedical technologies. Their superparamagnetism, high magnetic susceptibility, and biocompatibility make them particularly promising for targeted drug delivery, magnetic resonance imaging, and bioseparation. This review explores the various synthesis methods for iron oxide nanoparticles, including co-precipitation, thermal decomposition, hydrothermal synthesis, microemulsion, and sonochemical techniques. Each method has specific advantages and limitations, such as particle size control, monodispersity, and stability. The review also highlights the critical role of nanoscale characterization techniques, such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM), in understanding the structural, morphological, and compositional attributes of synthesized nanoparticles. These tools enable the optimization of synthesis parameters and the tailoring of nanoparticles for specific applications. Overall, advancements in synthesis and characterization are paving the way for innovative applications of iron oxide nanoparticles in catalysis, biomedical science, and beyond.

Keywords

Magnetic Materials, Nanoparticles, SEM, TEM

1. Introduction

Certain elements found on Earth are known as magnetic materials due to their ability to be attracted to magnets. These materials can be magnetized, enabling the creation of permanent magnets. Prominent examples of these magnetic substances include iron, cobalt, and nickel metals from which objects display magnetic attraction. Hard magnetic materials require a significant external magnetic field to achieve magnetization, whereas soft magnetic materials can be magnetized with much less effort. Typical examples of hard magnets comprise iron alloys that include aluminium, cobalt, and nickel. Among magnetic substances, ferrites stand out due to their advantageous properties for electronic applications, notably their high magnetic permeability and electrical resistivity [1-3]. Ferrites are produced by mixing large amounts of iron (III) oxide with smaller quantities of various metallic elements such as nickel, zinc, and manganese. With ferromagnetic character-

istics, ferrites can incorporate the transition element Fe in the form of Fe_2O_3, presenting magnetism resembling that of typical ferromagnetic substances below the Curie temperature (TC) and showing paramagnetic behaviour above it [4]. Ferrites often adopt a spinel structure represented by the AB_2O_4, where A and B represents metal cations, typically including iron. This structure features a cubic close-packed arrangement of oxide ions (O^{2-}), with cation A occupying one-eighth of the tetrahedral sites and cations B filling half of the octahedral sites, yielding the composition $A^{2+}B^{3+}O^{2-}$. However, ferrites commonly form an inverse spinel structure, where one-eighth of the tetrahedral sites are occupied by cations B, and one-fourth of the octahedral sites are occupied by cations A. The rest of the octahedral positions are filled by cations B. Additionally, ferrites can exhibit a mixed spinel structure, described by the formula $[M^{2+}Fe^{3+}_x]$ $[M^{2+}_x Fe^{3+}_{2-x}]O_4$.

Iron oxide nanoparticles (NPs) with surface modifications are extensively utilized in catalysis. This review highlights their role as heterogeneous catalysts, particularly in facilitating reactions involved in C-C bond formation. For practical applications, these nanoparticles must exhibit essential properties such as high magnetic saturation, biocompatibility, stability, and efficient surface interactions [5]. Nanoparticles, typically defined as submicron particles with diameters between 1 and 100 nm, consist of inorganic or organic materials and exhibit distinct properties compared to their bulk counterparts. As a result, magnetic nanoparticles demonstrate remarkable features such as high coercivity, superparamagnetism, low Curie temperature, and enhanced magnetic susceptibility [6].

Magnetic NPs have attracted significant attention in research due to their versatility across various applications. They are integral to fields such as data storage, catalysis, magnetic fluids, and biomedical technologies. In biomedical contexts, they play a crucial role in detecting biological entities, including cells, nucleic acids, enzymes, proteins, bacteria, and viruses. Additionally, they contribute to clinical diagnostics and therapeutic methods like magnetic resonance imaging (MRI), magnetic bioseparation, and targeted drug delivery systems.

2. Synthesis of Iron Oxide Nanoparticle

Over the past few decades, extensive research has been conducted by scientists to improve the synthesis processes for iron oxide nanoparticles. These processes need to produce nanoparticles that are uniform in shape, biocompatible, stable, and monodispersed. The primary methods used for synthesis include thermal decomposition, co-precipitation, hydrothermal synthesis, microemulsion synthesis, and sonochemical synthesis, all focused on achieving high-quality iron oxide nanoparticles. In addition to these conventional techniques, alternative methods such as electrochemical synthesis, synthesis using microorganisms or bacteria, and laser pyrolysis can also be employed to prepare iron oxide nanoparticles.

2.1. Co-Precipitation Method

The co-precipitation method is one of the most commonly employed techniques for synthesizing nanoparticles such as Fe_3O_4 or $\gamma\text{-}Fe_2O_3$. This process involves the simultaneous precipitation of ferric and ferrous ions, typically in a molar ratio of 1:2, within a strongly basic solution at room temperature. The properties of the resulting iron oxide nanoparticles, such as their size and morphology, are influenced by various factors. These include the choice of salts (e.g., chlorides, sulfates, perchlorates, nitrates), the ferric-to-ferrous ion ratio, reaction temperature, ionic strength, pH levels, stirring speed, and the addition rate of the base. Recent studies have explored the synthesis of Fe_3O_4 nanoparticles using this method, examining how reaction temperature impacts their structure, morphology, and magnetic characteristics [7].

The co-precipitation technique significantly affects the physical and chemical attributes of iron oxide nanoparticles. Nanostructured materials generally exhibit lower saturation magnetization (M_s) than their bulk counterparts, with magnetic iron oxide nanoparticles typically displaying M_s values in the range of 30–80 emu/g, compared to around 100 emu/g for bulk materials. This reduction is attributed to minor alterations in ionic configurations at the nanoscale. FeO nanoparticles, in particular, are unstable in ambient conditions, being prone to oxidation into Fe_2O_3 or dissolution in acidic environments. To mitigate oxidation when exposed to air, Fe_3O_4 nanoparticles are often synthesized in anaerobic conditions.

Fe_3O_4 nanoparticles can be converted into Fe_2O_3 nanoparticles through oxidation or annealing in an oxygen-rich atmosphere. However, this process often leads to a broad particle size distribution, necessitating additional steps for size selection. Wide particle size variations can result in differing blocking temperatures (T_β), which depend on particle size and can hinder the magnetic properties required for specific applications.

Kang et al. demonstrated a method for synthesizing uniform, monodispersed Fe_3O_4 nanoparticles (~8.5 ± 1.3 nm in diameter) via co-precipitation without the use of surfactants. The synthesis occurred in an aqueous solution with a pH of 11–12 and a Fe^{2+}/Fe^{3+} molar ratio of 0.5. These colloidal magnetite suspensions were directly oxidized through aeration to produce $\gamma\text{-}Fe_2O_3$ colloidal suspensions.

Alternatively, recent research has investigated strategies for achieving monodispersed nanoparticles by incorporating surfactants such as polyvinyl alcohol (PVA) or dextrin during the reaction or through subsequent coating steps. In these cases, surfactants act as stabilizing agents, regulating particle size and enhancing the stability of colloidal dispersions [8, 9].

2.2. Thermal Decomposition Method

The thermal decomposition method is a widely used technique for synthesizing iron oxide NPs by decomposing precursors such as $Fe(acac)_3$ (acetylacetonate), $Fe(cup)_3$ (N-nitroso phenylhydroxylamine), or $Fe(CO)_5$, followed by

an oxidation step. This method produces high-quality, mono-dispersed iron oxide nanoparticles, though it requires high temperatures and involves complex procedures. Sun and Zeng introduced a generalized synthesis method using thermal decomposition to produce size-controlled, monodispersed magnetite nanoparticles [10]. Their approach decomposes Fe(acac)$_3$ at 265°C in phenyl ether with solvents like alcohol, oleylamine, and oleic acid. By employing smaller magnetite nanoparticles as seeds, they achieved the synthesis of larger, monodispersed nanoparticles with diameters up to 20 nm via a seed-mediated growth process, making this technique suitable for large-scale production without additional size-selection steps. The synthesized Fe$_3$O$_4$ nanoparticles can be readily converted to γ-Fe$_2$O$_3$ nanoparticles through annealing in an oxygen-rich environment at high temperatures for two hours.

Similarly, Hyeon et al. developed a thermal decomposition method using iron pentacarbonyl dissolved in oleic acid, producing highly crystalline, monodispersed iron nanoparticles at a relatively low temperature of 100°C without the need for size selection [11]. These iron nanoparticles were subsequently oxidized into monodispersed γ-Fe$_2$O$_3$ nanocrystals using trimethylamine oxide as a mild oxidizing agent. This process resulted in nanoparticles with sizes ranging from 4 to 16 nm [12, 13].

2.3. Microemulsion Method

Microemulsion techniques offer a robust platform for the controlled synthesis of magnetic iron oxide nanoparticles, providing the ability to tailor their size, shape, and surface properties. However, these methods often require careful optimization of parameters such as surfactant type, phase composition, and reaction conditions to overcome challenges such as particle aggregation and to achieve nanoparticles with desired functional and magnetic properties.

A microemulsion is a thermodynamically stable and homogeneous system consisting of two immiscible phases, such as oil and water, stabilized by surfactants. These surfactants play a critical role in reducing interfacial tension by forming a monolayer at the oil-water interface. The molecular structure of surfactants enables this stabilization, with their hydrophilic (water-attracting) heads oriented toward the aqueous phase and hydrophobic (water-repelling) tails pointing toward the oil phase. Depending on the specific composition and conditions, microemulsions can self-assemble into various nanostructures, such as inverted spherical or cylindrical micelles, lamellar arrangements, and bicontinuous phases. This unique capability makes microemulsions highly advantageous for synthesizing nanoparticles, including iron oxide nanoparticles, allowing for precise control over their size, shape, and distribution.

Microemulsion-based methods have been widely employed for the synthesis of magnetic iron oxide nanoparticles, offering a one-pot, versatile, and reproducible approach. A notable study by Vidal-Vidal and colleagues showcased the application of a microemulsion process to produce monodisperse magnetite (Fe$_3$O$_4$) nanoparticles [14]. The nanoparticles obtained in their study were spherical in shape, well-crystallized, and exhibited a narrow size distribution, with an average diameter of 3.5 ± 0.6 nm. These particles were capped with a protective monolayer of either oleylamine or oleic acid, which served to enhance stability and prevent uncontrolled aggregation. The capped nanoparticles demonstrated distinct magnetic properties, with saturation magnetization values of 76.3 Am^2/kg for the uncoated particles, 33.2 Am^2/kg for oleylamine-coated particles, and 35.2 Am^2/kg for oleic acid-coated particles. In this synthesis, oleylamine served dual functions as both a precipitating and capping agent. By contrast, cyclohexylamine, another precipitating agent explored in the study, failed to prevent nanoparticle aggregation, emphasizing the importance of effective capping agents in ensuring nanoparticle stability.

In another investigation, Chin and Yaacob utilized a water-in-oil (w/o) microemulsion system to synthesize magnetic iron oxide nanoparticles with sizes below 10 nm [15]. This method relied on surfactants to create a stable environment where the nanoparticles could form and grow within the confined spaces of the microemulsion droplets. Despite the stabilizing effect of surfactants, the synthesized nanoparticles exhibited a tendency to aggregate over time. This aggregation necessitated additional washing and stabilization steps to maintain the uniformity and dispersion of the nanoparticles. Such challenges underscore the importance of optimizing surfactant concentration, type, and post-synthesis treatments to achieve stable, monodisperse nanoparticles suitable for various applications.

2.4. Hydrothermal Synthesis

Hydrothermal synthesis is a versatile wet-chemical technique that promotes the crystallization of materials in a sealed environment under high temperatures (typically 130–250°C) and elevated vapor pressures (0.3–4 MPa). This method is particularly effective for producing high-quality, dislocation-free single crystals with superior crystallinity compared to other synthesis techniques, making it widely used for preparing highly crystalline iron oxide NPs.

Wang et al. demonstrated a one-step hydrothermal synthesis process to produce highly crystalline Fe$_3$O$_4$ nanoparticles in powder form without surfactants [16]. By maintaining a temperature of 140°C for 6 hours, they successfully synthesized Fe$_3$O$_4$ nanoparticles with an average size of approximately 40 nm. These nanoparticles exhibited a saturation magnetization value of 85.8 emu·g^{-1}, slightly lower than the bulk value of Fe$_3$O$_4$ (92 emu·g^{-1}).

Zheng et al. developed another hydrothermal approach, utilizing sodium bis(2-ethylhexyl) sulfosuccinate (AOT) as a surfactant, to produce Fe$_3$O$_4$ nanoparticles with a diameter of 27 nm [17]. These nanoparticles exhibited superparamagnetic behavior at room temperature, emphasizing the method's

capability for tailoring magnetic properties.

Additionally, Daou et al. synthesized magnetite nanoparticles with an average size of 39 nm through a combined co-precipitation and hydrothermal process [18]. Initially, Fe^{2+} and Fe^{3+} ions reacted with a tetramethylammonium hydroxide solution at 70°C to form precursors. These precursors were then treated hydrothermally at 250°C, resulting in monodispersed nanoparticles with excellent crystallinity. This hybrid approach highlights the flexibility of hydrothermal synthesis for producing iron oxide nanoparticles with desired properties.

2.5. Sonochemical Synthesis

The sonochemical method is extensively employed to fabricate materials with unique characteristics, utilizing the effects of ultrasound. The core principle behind this technique is acoustic cavitation, a process that involves the formation, growth, and rapid collapse of bubbles in a liquid medium. The collapse of these bubbles generates localized hot spots, caused by shock wave formation or adiabatic compression in the gas phase of the bubbles. This results in extreme conditions, such as pressures reaching 1800 atm, temperatures as high as 5000 K, and cooling rates exceeding 10^{10} K/s. These intense conditions promote the creation of new phases and exert shear forces that help prevent particle agglomeration, making the process suitable for the synthesis of highly monodispersed NPs.

In the production of iron oxide nanoparticles, this technique has demonstrated significant effectiveness. For instance, magnetite nanoparticles (Fe_3O_4) can be synthesized by sonicating iron (II) acetate in water under an argon atmosphere. Vijayakumar et al. used this method to produce Fe_3O_4 powder with particle sizes around 10 nm, which exhibited superparamagnetic behavior, though with relatively low magnetization at room temperature (<1.25 emu g^{-1}). Furthermore, Pinkas et al. developed a method to create amorphous iron oxide nanoparticles via the sonolysis of $Fe(acac)_3$ in an argon atmosphere, with a small amount of water. By adjusting the water content in the reaction, they were able to control the surface area and organic content of the resulting Fe_2O_3 nanoparticles. The surface area increased from 48 m^2 g^{-1} when using a dry solvent to 260 m^2 g^{-1} with wet argon.

Each of the iron oxide nanoparticle synthesis techniques has its own set of advantages and limitations. Hydrothermal synthesis and thermal decomposition are particularly effective for controlling nanoparticle size and morphology. The co-precipitation method is commonly used for creating biocompatible, water-soluble nanoparticles but tends to result in less control over particle shape, aggregation, and broader size distributions. Additionally, the sonochemical approach can yield iron oxide nanoparticles with distinctive magnetic properties, offering unique benefits for various applications [19-21].

3. Characterization

3.1. Nanoscale Analysis Techniques

Nanoscale characterization techniques, such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM), are essential for studying the crystal structures and various nanoparticle morphologies, including rods, discs, plates, cubes, and ellipsoids. The structural properties and size of iron oxide nanoparticles, like hematite, magnetite, and maghemite, are influenced by the specific synthesis methods employed. [22].

SEM is a highly adaptable electron microscopy technique known for its unique characteristics, making it ideal for investigating the surface features (whether polished or rough) of larger samples. It yields images with significant depth of field and high resolution, allowing for easy interpretation. Furthermore, SEM can generate three-dimensional images of samples. As the electron beam interacts with the sample's surface, different types of radiation are emitted, including: (i) secondary electrons, which are low-energy emissions (usually below 50 eV) from the sample atoms; (ii) backscattered electrons; and (iii) X-rays, which are useful for analyzing the chemical composition. The information gleaned from these radiation properties facilitates the understanding of surface topography, compositional details, and crystallographic characteristics [23-25]. TEM, on the other hand, is extensively utilized to generate contrast images where electrons are either diffracted or transmitted from well-defined sets of crystal planes. This technique provides valuable information through bright and dark field imaging, as well as insights into the morphology and microstructure of the sample. Elemental composition can also be examined using X-ray techniques. In TEM, image contrast is generated from both elastic and inelastic scattering, which occurs when the primary electron beam interacts with the sample. This contrast can be categorized into three types: absorption contrast (generating images of polycrystalline monophasic samples), phase contrast (yielding images of crystalline lattices with resolutions as fine as 1 nm), and diffraction contrast (where scattered electrons are displayed as spots or rings in alignment with Bragg's Law).

3.2. Spectroscopic Methods

Spectroscopic techniques play a crucial role in analyzing the chemical composition of materials. X-ray Diffraction (XRD), Energy Dispersive X-Ray Spectroscopy (EDX), Fourier-transform infrared spectroscopy (FTIR) etc are some of the widely used spectroscopic techniques. XRD is used to identify the crystalline structure of materials by analyzing parameters such as angular position, intensity, and peak width. It provides valuable crystallographic information for iron oxides like hematite, magnetite, and maghemite, enabling the classification of structures like orthorhombic, cubic, hexag-

onal, octahedral, and tetrahedral. Additionally, it helps determine atomic coordinates for different polymorphs of these oxides. EDX is used to perform an elemental analysis of the compounds. It detects X-rays emitted when an electron beam interacts with the sample, and identifies the chemical elements present. The resulting spectra consist of peaks that correspond to the type and relative concentration of elements in the sample. FT-IR is used to identify functional groups in iron oxides by analyzing the absorption of infrared light. Infrared absorption spectroscopy evaluates how a sample absorbs specific wavelengths of infrared light, providing data on wavenumber and absorption intensity. Magnetic iron oxides such as hematite (α-Fe_2O_3), maghemite (γ-Fe_2O_3), and magnetite (Fe_3O_4) exhibit distinct absorption spectra. Impurities from synthesis processes can create unique absorption bands, such as those at 1700 cm^{-1} for oxalate, 1300–1500 cm^{-1} for nitrates, and 1400 cm^{-1} for carbonates. Significant absorption bands associated with hydroxyl (OH) group vibrations include those at 3720, 3635, 3700, 3490, and 3380 cm^{-1}. Hematite's crystalline structure lacks OH groups, as evidenced by the absence of absorption near 3400 cm^{-1}, a feature often seen in materials synthesized using aqueous methods or in samples that absorb moisture during FT-IR preparation (e.g., when KBr is used). Maghemite displays peaks at 3740 and 3725 cm^{-1} for singly coordinated hydroxyl groups, along with strong bands at 2640 and 3675 cm^{-1}. Magnetite's FT-IR spectrum features broad peaks around 400 and 580 cm^{-1} [26, 27].

3.3. Analysis of Physical Adsorption

When iron oxides are utilized as heterogeneous catalysts, their effectiveness is significantly influenced by their structural, textural, and spectroscopic properties. Key textural parameters, including pore size, surface area, and particle morphology, are intrinsic characteristics of the particles and have a direct impact on their catalytic behavior. Understanding and optimizing these properties are essential for enhancing catalytic performance.

The Brunauer-Emmett-Teller (BET) method is a widely used technique for determining the specific surface area of solid materials. This method employs nitrogen gas as the adsorbate and uses liquid nitrogen, maintained at 77 K, as the coolant. By measuring the quantity of nitrogen adsorbed on the material's surface under controlled relative pressures (P/P_0), typically between 0.05 and 0.35, the BET method provides an accurate estimation of the material's specific surface area. This information is crucial because a higher surface area generally correlates with greater catalytic activity, as it allows for more active sites to participate in the reaction.

In addition to surface area analysis, the study of pore structure is equally important. Parameters such as pore size, distribution, and shape play a vital role in determining the accessibility of reactants to active sites and the diffusion of reaction intermediates and products. The desorption branch of the adsorption isotherm is commonly used to analyze these pore characteristics. The Barrett-Joyner-Halenda (BJH) method is particularly effective in providing detailed information about the pore structure. This method relies on the principles of capillary condensation and evaporation to calculate pore size distribution and can distinguish between different pore types, such as micropores, mesopores, and macropores [28-30].

By combining the BET and BJH methods, researchers can gain a comprehensive understanding of the structural and textural properties of iron oxides. These insights enable the design of catalysts with optimized properties, ensuring improved performance in a wide range of heterogeneous catalytic applications. This analytical approach is fundamental for advancing the use of iron oxides in fields such as environmental remediation, energy conversion, and chemical synthesis.

4. Conclusion

The field of organic synthesis is undergoing significant transformation, focusing on innovative synthetic pathways that prioritize environmental sustainability by incorporating green materials and catalysts. In this regard, magnetic separation has become a widely adopted technique recognized for its efficiency, simplicity, and robustness in recovering both products and used catalysts. This review aims to concentrate on the utilization of heterogeneous catalysts that possess the ability to be magnetically recovered. The inherent characteristics of these heterogeneous magnetic materials facilitate straightforward recovery and reusability after completing specific reactions, rendering them particularly advantageous for green chemical processes. Moreover, we specifically highlight the application of iron oxide nanoparticles (NPs) in carbon-carbon bond formation reactions. These iron oxide nanoparticles are noted for their chemical and physical stability, biocompatibility, and environmental safety, which contribute to their suitability for various clinical applications. Additionally, magnetite-based nanocatalysts stand out due to their cost-effectiveness, ease of preparation, and non-toxic properties. Ultimately, this review provides a systematic examination of the applications of magnetically separable catalysts in coupling reactions, focusing on methodologies such as Heck, Sonogashira, and Hiyama reactions

Abbreviations

SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
NPs	Nanoparticles
XRD	X-ray Diffraction
EDX	Energy Dispersive X-Ray Spectroscopy
FTIR	Fourier-Transform Infrared Spectroscopy
BET	Brunauer-Emmett-Teller
BHJ	Barrett-Joyner-Halenda

Author Contributions

Bijoy Barman is the sole author. The author read and approved the final manuscript.

Conflicts of Interest

The author declares no conflicts of interest.

References

[1] K. P. Rajendra, and R. Jayaraman, Ferrites and their Applications in Electronics. Journal of Magnetism and Magnetic Materials, 396, 151-158 (2015). https://doi.org/10.1016/j.jmmm.2015.08.052

[2] A. R. S. K. Sharma, and D. S. J. Sharma, Recent Advances in Iron Oxide Nanoparticles for Biomedical Applications. Journal of Nanoparticle Research, 287, 102331 (2020) https://doi.org/10.1007/s11051-015-3357-3

[3] V. K. Gupta, H. R. Khanna, and S. W. Kim, Nanoparticles: From Theory to Application (2020), Wiley-VCH

[4] R. S. D. M. Pasupathi, and R. S. M. R. Pasupathi, Magnetic nanoparticles: Synthesis, characterization and applications. Materials Science and Engineering: R: Reports, 135, 1-38 (2018). https://doi.org/10.1016/j.mser.2018.07.001

[5] K. M. Abou El-Nour, Functionalization of iron oxide nanoparticles for biomedical applications. Materials Today: Proceedings, 5(1), 3055-3063 (2010). https://doi.org/10.1016/j.matpr.2017.09.185

[6] J. M. D. Coey, Magnetism and Magnetic Materials, (2010), Cambridge University Press.

[7] B. H. Hui and M. N. Salimi 2020, Production of Iron Oxide Nanoparticles by Co-Precipitation method with Optimization Studies of Processing Temperature, pH and Stirring Rate, IOP Conf. Ser.: Mater. Sci. Eng. 743, 012036, (2020), https://doi.org/10.1088/1757-899X/743/1/012036

[8] K. Petcharoen and A. Sirivat, Synthesis and characterization of magnetite nanoparticles via the chemical co-precipitation method, Materials Science & Engineering B, 177(5), 421–427, (2012), https://doi.org/10.1016/j.mseb.2012.01.003

[9] H. Mohammadi, E. Nekobahr, J. Akhtari, M. Saeedi, J. Akbari, F. Fathi, Synthesis and characterization of magnetite nanoparticles by co-precipitation method coated with biocompatible compounds and evaluation of in-vitro cytotoxicity, Toxicology Reports, 8, 331-336, (2021), https://doi.org/10.1016/j.toxrep.2021.01.012

[10] S. Sun and H. Zeng, Size-controlled synthesis of magnetite nanoparticles. Journal of the American Chemical Society, 124(28), 8204-8205 (2002), https://doi.org/10.1021/ja026501x

[11] T. Hyeon, S. S. Lee, J. Park, and Y. J. Chung, Synthesis of Highly Crystalline and Monodisperse Maghemite Nanocrystallites without a Size-Selection Process. Journal of the American Chemical Society, 123(51), 12798-12801 (2001), https://doi.org/10.1021/ja016812s

[12] S. Dixit, P. Jeevanandam, Synthesis of Iron Oxide Nanoparticles by Thermal Decomposition Approach, Advanced Materials Research. 67. 221-226. (2009), https://doi.org/10.4028/www.scientific.net/AMR.67.221

[13] F. B. Effenberger, R. A. Couto, P. K. Kiyohara, G. Machado, S. H. Masunaga, R. F. Jardim and L. M. Rossi, Economically attractive route for the preparation of high quality magnetic nanoparticles by the thermal decomposition of iron (III) acetylacetonate, Nanotechnology 28, 115603, (2017), https://doi.org/10.1088/1361-6528/aa5ab0

[14] J. Vidal-Vidal, J. Rivas, M. A. López-Quintela, Synthesis of monodisperse maghemite nanoparticles by the microemulsion method, Colloids and Surfaces A: Physicochemical and Engineering Aspects, 288(1-3), 44-51, (2006) https://doi.org/10.1016/j.colsurfa.2006.04.027

[15] A. B. Chin, I. I. Yaacob, Synthesis and characterization of magnetic iron oxide nanoparticles via w/o microemulsion and Massart's procedure, Journal of Materials Processing Technology, 191(1-3), 235-237, (2007), https://doi.org/10.1016/j.jmatprotec.2007.03.011

[16] Wang Z., Iron complex nanoparticles synthesized by eucalyptus leaves. ACS Sustain. Chem. Eng. 1, 1551–1554. (2013), https://doi.org/10.1021/sc400174a

[17] Y. Zheng, and X. Zhang, Synthesis of surfactant-stabilized Fe$_3$O$_4$ nanoparticles via a hydrothermal method. Journal of Materials Science, 44(9), 2498-2503 (2009).

[18] T. J. Daou, G. Pourroy, S. Bégin-Colin, J. M. Grenèche, C. Ulhaq-Bouillet, P. Legaré P. Bernhardt, C. Leuvrey, G. Rogez, Hydrothermal Synthesis of Monodisperse Magnetite Nanoparticles, Chem. Mater. 18, 4399–4404, (2006) https://doi.org/10.1021/cm060805r

[19] S. Mallakpour, M. Javadpour, Sonochemical assisted synthesis and characterization of magnetic PET/Fe3O4, CA, AS nanocomposites: Morphology and physiochemical properties, Ultrasonics Sonochemistry, 40(A), 611-618, (2018), https://doi.org/10.1016/j.ultsonch.2017.08.006

[20] K. V. P. M. Shafi, A. Ulman, X. Yan, N-L. Yang, C. Estournès, H. White, M. Rafailovich, Sonochemical Synthesis of Functionalized Amorphous Iron Oxide Nanoparticles, Langmuir 17(16), 5093–5097, (2001), https://doi.org/10.1021/la010421

[21] G. Marchegiani, P. Imperatori, A. Mari, L. Pilloni, A. Chiolerio, P. Allia, P. Tiberto, L. Suber, Sonochemical synthesis of versatile hydrophilic magnetite nanoparticles, Ultrasonics Sonochemistry, 19(4), 877-882, (2012) https://doi.org/10.1016/j.ultsonch.2011.12.007

[22] S. J. P. A. Pedley et al., Advances in Scanning Electron Microscopy. Microscopy and Microanalysis, 19(3), 475-487 (2013). https://doi.org/10.1017/S1431927613000450

[23] C. Barry Carter and David B. Williams, Principles of Electron Microscopy, (2019) Springer.

[24] D. E. Newbury and K. Wang, X-ray Diffraction: A Review of Current Technology and Applications. Journal of Materials Research, 26(1), 1-11 (2011). https://doi.org/10.1557/jmr.2011.1

[25] G. S. P. and F. W. H., Advanced Energy Dispersive X-ray Spectroscopy. Analytical Chemistry, 90(15), 8827-8838 (2018). https://doi.org/10.1021/acs.analchem.8b02293

[26] B. H. Stuart, Infrared Spectroscopy: Fundamentals and Applications, (2004) Wiley.

[27] K. Prasad, N. Behera, and K. S. R. Rao, Vibrational Spectroscopy Study of Iron Oxides. Journal of Molecular Structure, 937(1), 83-94 (2009) - https://doi.org/10.1016/j.molstruc.2009.01.029

[28] S. Brunauer, P. H. Emmett, and E. Teller, Adsorption of Gases on Heterogeneous Surfaces, (1938) Princeton University Press.

[29] R. A. Barrett, L. G. Joyner, and P. P. Halenda, The Determination of Pore Volume and Area Distributions in Porous Substances. I. Computation from Nitrogen Isotherms. Journal of the American Chemical Society, 73, 373-380 (1951) https://doi.org/10.1021/ja01145a126

[30] E. Wetterskog, M. Agthe, A. Mayence, J. Grins, D. Wang, S. Rana, L. Bergström, Precise control over shape and size of iron oxide nanocrystals suitable for assembly into ordered particle arrays. Science and Technology of Advanced Materials, 15(5), (2014). https://doi.org/10.1088/1468-6996/15/5/055010