



A review of Structure, Properties, and Chemical Synthesis of Magnetite Nanoparticles

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Abstract

Extensive studies were devoted to iron oxide nanoparticles (IONPs), in recent years. Iron oxides are chemical compounds that have various polymorphic forms, including maghemite (γ -Fe₂O₃), magnetite (Fe₃O₄), and Hematite (α -Fe₂O₃). Among them, the most important studied is magnetite (Fe₃O₄) due to its low cost and low toxicity, besides its unique magnetic and physicochemical characteristics which qualified it for use in various applications such as biomedical and technological applications. Magnetic particles should be small and have a narrow size distribution for these applications. The smaller the size of the iron oxide particles, the greater their reactivity and biodegradability. In this review, we display summary information of magnetite (Fe₃O₄) nanoparticles in terms of structure, characteristics, and preparation methods. Because the prepared strategy has been proven to be critical for preferable control of the particle size and shape, in addition, to producing monodispersed magnetite (Fe₃O₄) nanoparticles with a direct effect on their characteristics and applications, special attention will be placed on chemical preparation techniques including Hydrothermal synthesis, Co-precipitation technique, Sol-Gel process, and thermal decomposition method. This review offers specific information for selecting appropriate synthetic methods for obtaining appropriate sizes, shapes, and magnetic properties of magnetite (Fe₃O₄) nanoparticles (NPs) for target applications.

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1. Introduction

When compared to macro-sized particles, nanoparticles (NPs) have a higher surface area. At the atomic level (1–100) nm, NPs are referred to as controlled or manipulated particles. They exhibit size-related characteristics that vary significantly from bulk materials. When compared to similar materials in bulk dimensions, these structures have distinct and desirable chemical and physical properties, such as a special surface area and optical, magnetic, electrical, thermal, and mechanical behavior [1- 6]. Magnetic nanoparticles (MNPs) contain numerous distinct magnetic properties, including high magnetic susceptibility, high coercivity, superparamagnetic, low Curie temperature, etc. [7]. MNPs have piqued the attention of researchers due to their fascinating properties and wide

range of diverse applications in high-density data storage, ferrofluids, and as catalysts. In bioapplications, containing detection of biological entities (virus, bacterial, enzyme, nucleic acids, cell, protein, etc.) and magnetic bioseparation [7]. In addition, MNPs have been employed to create heat to treat hyperthermia, produce contrast influences for magnetic imaging, and dominate targeted drug delivery remotely [8]. For a variety of factors, iron oxide nanoparticles have been identified as the best candidate. (1) abundance, (2) simple preparation, (3) simple access to several oxidation states and polymorphs, (4) a diverse range of electrical and magnetic properties, and (5) Iron oxide nanoparticles are an appropriate prototype of functional material with a varied spectrum of electrical and magnetic properties due to their low toxicity and spontaneous elimination [9]. In addition, due to their high biocompatibility, iron oxide nanoparticles are promising nanomaterials. The biocompatibility of iron oxide nanoparticles is the primary factor propelling significant research efforts to commercialize these nanoparticles for use in sophisticated applications of medical technology [8, 10, 11]. Iron oxide can be found in variations of forms in nature. The most prevalent are hematite (α -Fe₂O₃), maghemite (γ -Fe₂O₃), and magnetite (Fe₃O₄) [12, 13]. Magnetite (Fe₃O₄) is a promising candidate among known crystal polymorphs of iron (III) due to its biocompatibility and biodegradable activity [14]. Fe₃O₄ NPs exhibit either superparamagnetic (if the size is under 15 nm) or ferromagnetic behavior [8]. Magnetite is a naturally occurring mineral that has been greatly utilized in biological applications inclusive, magnetic separation, magnetic drug delivery, magnetic, resonance imaging, and magnetic hyperthermia [8, 15- 20]. The physical, chemical, and biological approaches are currently the three most significant reported routes for the creation of Fe₃O₄ nanoparticles [21, 22]. Chemical methods have an advantage over physical and biological ones when it comes to creating new materials with higher chemical homogeneity by combining different precursors and carefully regulating the size, shape, and content of nanoparticles. The chemical pathway also saves time and money because it does not require expensive tools or ingredients, making it a good method for manufacturing nanoparticles. Yet, the chemical method has significant downsides, such as the production of excess intermediates and contaminants and the possibility of colloidal agglomeration occurring during the synthesis process [23]. In this paper, we will focus on the influence of the chemical synthesis on the geometries, sizes, and thus magnetic characteristics of magnetite NPs, in addition, to the structure and summary of the properties of Fe₃O₄ nanoparticles.

2. Iron Oxides

There are eight known iron oxides [24]. Because of their polymorphism, which involves temperature-induced phase transitions, Hematite, maghemite, and magnetite are so widespread candidates among these iron oxides and each one has distinct catalytic, magnetic, biochemical, and characteristics that make them suitable for a variety of biomedical and technical applications [25].

2.1. Hematite (α -Fe₂O₃)

With an antiferromagnetic order below Néel temperature and a corundum crystal structure, α -Fe₂O₃ is the most stable iron oxide phase (955 K). Two-thirds of the octahedral sites limited through the roughly ideal hexagonal close-packed Oxygen lattice are occupied by Fe³⁺ ions, as revealed in Figure 1(a). Due to its inexpensive and high corrosion resistance, hematite (α -Fe₂O₃) is commonly utilized in gas sensors, pigments, and catalysts, and as a source used for the preparation of magnetite and maghemite. Hematite is a 2.1 eV band gap n-type semiconductor under the circumstances of the environment [25- 27].

2.2. Magnetite (Fe₃O₄)

Magnetite has a face-centered cubic with an inverse spinel structure, set up on thirty-two Oxygen ions and packed closely along the direction [28]. Magnetite includes both divalent and trivalent iron, unlike most other iron oxides. Fe²⁺ ions occupy 1/2 octahedral positions and the Fe³⁺ ions are distributed equally across the residual tetrahedral and octahedral positions. Fe³⁺ ions in A and B positions are antiferromagnetically coupled, whilst Fe²⁺ ions in the B position participate in macroscopic ferromagnetic characteristics, as displayed in Figure 1(b). Fe³⁺ ions within A and B positions are coupled antiferromagnetically while Fe²⁺ ions within the B position share macroscopic ferromagnetic characteristics. The divalent irons can be replaced partially or completely by another divalent ion (Zn, Mn, Co, etc). So, magnetite can be either p-type or n-type semiconductors. Due to its low band gap (0.1 eV), magnetite possesses the lowest resistivity among all iron oxides. Fe₃O₄ easily undergoes a phase transformation to maghemite at room temperature [25, 27].

2.3. Maghemite (γ -Fe₂O₃)

Maghemite has a cubic structure, each unit of $\gamma\text{-Fe}_2\text{O}_3$ has $21\frac{1}{3}$ Fe^{3+} ions, $2\frac{1}{3}$ vacancies, and 32O^{2-} ions. The O^{2-} ions form a cubic packed closely array, whilst the Fe^{3+} is spread between tetrahedral sites (8 Fe ions per unit cell) and octahedral positions (the residual iron ions and vacancies), as shown in Figure 1 (c). Maghemite is oxidized magnetite and a 2.0 eV bandgap n-type semiconductor. Maghemite with good dispersivity in aqueous media can be synthesized by an inexpensive technique. It is classified as a ferromagnetic oxide and possesses a spinel structure nearly to that of magnetite [25, 27].

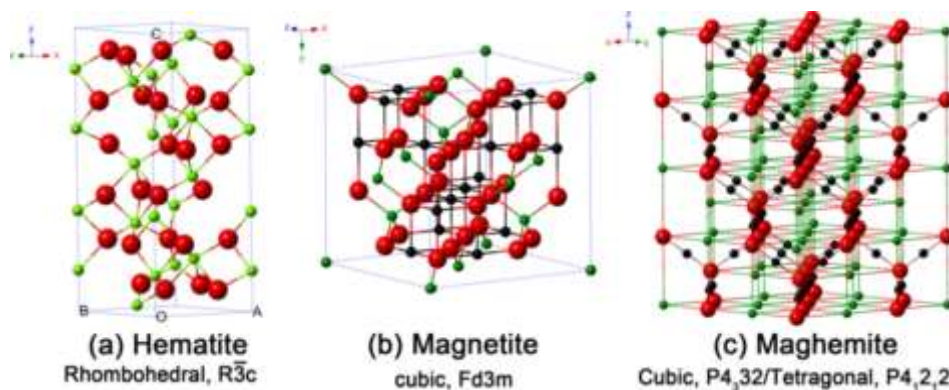


Figure 1: Crystal structure of the Hematite, Magnetite, and Maghemite (the black ball is Fe^{2+} , the green ball is Fe^{3+} and the red ball is O^{2-}) [25].

3. Properties of Magnetite (Fe_3O_4) Nanoparticles

The magnetic characteristics of Fe_3O_4 NPs are controlled via the size of the particle. When ferrimagnetic Fe_3O_4 NPs are enough small in size, they exhibit superparamagnetic characteristics with a great response to the magnetic field applied. Figure 2 depicts the transformation of ferrimagnetic to superparamagnetic characteristics, where the MNPs change to single-domain magnetism from multi-domain magnetism as their size decreases. The increase of coercivity to a maximum value is due to the reduction of size to a specific size called the critical diameter, D_s . At this point, the same direction is indicated by all of the magnetic spins, improving the magnetic property, and MNPs are typically hard to demagnetize due to their high coercivity. More decreasing size rapidly reduces the coercivity value until it equals zero, at which point the NPs are said to be in a superparamagnetic state. Generally, Fe_3O_4 NPs with diameters less than 20 nm have superparamagnetic properties [8, 23].

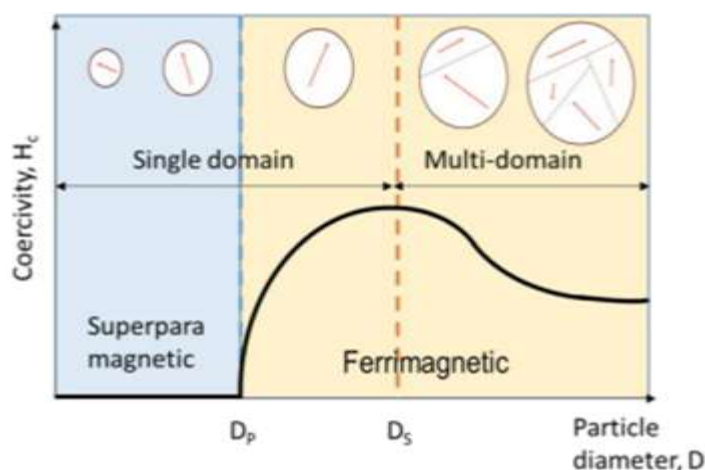


Figure 2: Relation between coercivity, H_c , and magnetic particle diameter, D [23].

As displayed in Figure 3, superparamagnetic Fe_3O_4 NPs differ from ferrimagnetic particles in that they lack coercive force and hysteresis loops because of single-domain magnetism, allowing them to be magnetized just in the existence of an external magnetic field. As a result, employing an external magnetic field to control these superparamagnetic Fe_3O_4 nanoparticles is simple. Superparamagnetic nanoparticles exhibit a stronger and quicker magnetic response to an external magnetic field, which is also worth noticing [23].

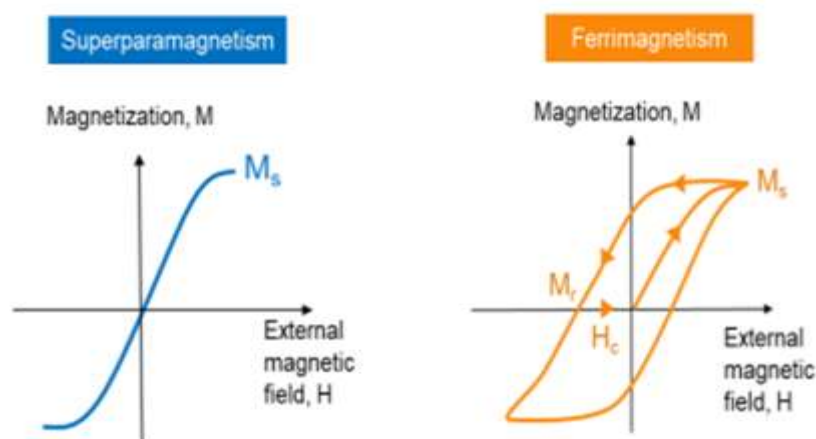


Figure 3: Magnetization vs. applied field (M–H) curve of the superparamagnetism (blue color) and ferrimagnetism (orange color) [23].

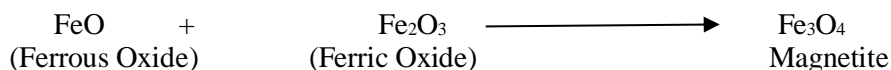
Table 1 displays the physical and magnetic properties of Fe₃O₄ NPs [24, 29, and 30]. In the biological field, the saturation magnetization values are the most important feature. High saturation magnetization values improve drug delivery to cancer cells, image projection resolution in MRI, and heat dissipation in MHT.

Table 1: Summary of the properties of Fe₃O₄ nanoparticles.

Property	Magnetite
Molecular formula	Fe ₃ O ₄
Type of magnetism	Ferrimagnetic
Density (g/cm ³)	5.18
Curie temperature (K)	850
Saturation magnetization (Ms) at 300K [emu/g]	92–100
Melting point (°C)	1583-1597
Standard Gibbs free energy of formation (ΔG _f ⁰) [kJ/mol]	-1012.6
Structural type	Inverse spinel
Crystallographic system	Cubic
Lattice parameter (nm)	a = 0.8396
Lattice angles	α = γ = β = 90
Band gap energy (Eg) [eV]	2.6
Color	Black
Formula units/unit cell	8
Hardness	5.5

4. Chemical Methods for Synthesis of Fe₃O₄ NPs

A typical magnetite preparation reaction is explained below by depicting the compound's chemical composition [29].



Much research has been developed in the last few decades to prepare iron oxide nanoparticles, and considerable studies have been published that describe efficient synthesis methods for producing stable, biocompatible, shape-controlled, and monodispersed iron oxide NPs. Hydrothermal synthesis [7], Co-precipitation [31], Sol-Gel, and thermal decomposition method [28], are all Chemical methods for producing high-quality magnetite NPs.

4.1. Hydrothermal Synthesis

These reactions take place in aqueous media in autoclaves or reactors where the pressure (generally between 0.3 and 4 MPa) and temperature can be adjusted (in general from 130 - 250°C. To achieve supersaturating,

hydrothermal procedures depend on the capability of water to hydrolyze and dehydrate metal salts at extreme conditions, as well as the very low solubility of the resultant metal oxides in water at these limits [7, 32]. Iron oxide NPs with appropriate shape and size control are technologically significant because of the powerful correlation between these parameters and magnetic characteristics [33]. The hydrothermal approach is environmentally friendly, inexpensive, and straightforward, and the reaction was carried out at relatively low temperatures. This technique controls particle size in crystallization and morphology via reaction time and temperature, allowing for the creation of high-quality nanoparticles [28, 34-38]. The hydrothermal method is sometimes used to prepare single crystal particles free of dislocation defects, and grains created in this technique may possess higher crystallinity than grains formed in other methods, implying that hydrothermal synthesis is more likely to produce iron oxide NPs with highly crystalline [7]. Many researchers successfully fabricated iron oxide NPs by hydrothermal method [39- 44]. In 2014 [40], conducted a comprehensive investigation of the influence of reaction time and temperature on particle size in this process. As stated by transmission electron microscopy examination, the size of the NPs increased from (14.5- 29.9) nm at reaction temperature raised from 100 – 180°C for twelve hours. At 180°C, the size of nanoparticles grows from (20.6 – 123.44) nm as the reaction time was raised from (1 – 48) h. This means that the reaction times had a more significant impact on the particle size than temperature. N. Gómez et al. [28], fabricated Fe₃O₄ NPs via a hydrothermal process. In addition, they investigated the influence of reaction temperature on the morphology, phase structure, particle size, and the shape of products. The X-ray diffraction pattern displayed that all the NPs were Fe₃O₄ in a pure magnetite phase. The produced NPs had uniform morphology with a high level of crystallinity at all temperatures, as noticed by TEM. Figure 4 revealed a TEM image of NPs prepared at 120°C. As a result, by elevating the temperature to 120°C, 140°C, and 160°C, it was feasible to create quasi-spheres, octahedrons, and cubes in the nanometric regime. The hydrothermal method was chosen by S. Ni, et al. [44], to produce well dispersed, well crystallized and, high purity Fe₃O₄ nanoparticles which can be easily obtained by this method. The product was obtained at 90 C for 24 h with an average diameter of approximately 160 nm in the presence of sodium sulfate. S. Ahmadi et al. [43] have used a facile hydrothermal technique to fabricate highly crystallite Fe₃O₄ nanocrystals. The calculated average crystallite sizes are 13.4, 20.8, and 22.8 nm for the magnetite formed at 100, 150, and 200°C, sequentially. It is suggested that elevated temperature is desirable for the preparation of bigger magnetite NPs. Both crystallite and average particle size of the magnetite NPs are good identical, showing the single crystal structure of the crystals. The formed magnetite nanocrystals have superparamagnetic behavior whereas the saturation magnetization, and the coercivity increment with the increment of the hydrothermal temperature. The increment of both the saturation magnetization and the coercivity of the magnetite nanocrystals can be referred to as the spin canting influence and the reduced ratio of the surface-to-volume of the nanocrystals. Figure 5 displays the hydrothermal synthesis of IONPs.

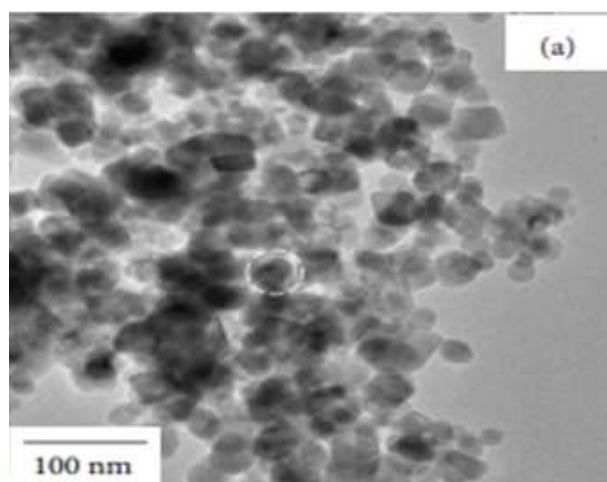


Figure 4: TEM image of the Fe₃O₄ NPs at 120°C [28].

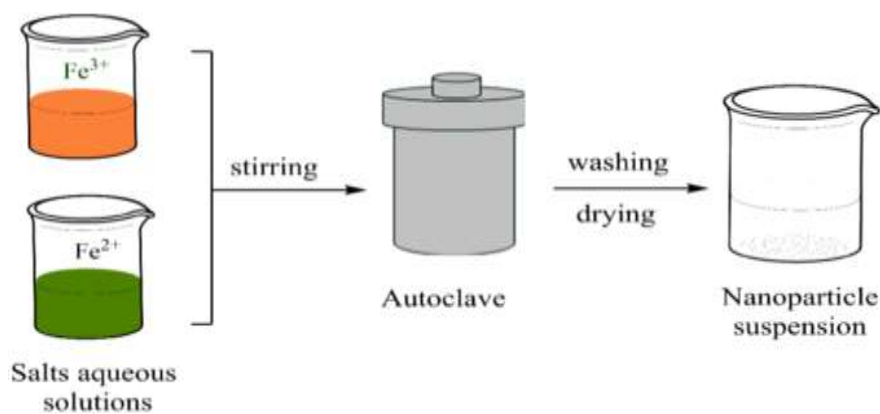


Figure 5: Hydrothermal synthesis of IONPs [45].

4.2. Co-precipitation

Co-precipitation is the most generally utilized method for producing Fe_3O_4 because of its advantages, which include an inexpensive and simple synthetic procedure, a high yield product with exceptional magnetic and crystal characteristics, and the use of an inorganic reactant. This process involves mixing ferric and ferrous ions in extremely basic solutions at elevated temperature or room temperature in a 2:1 molar ratio. The morphology and size of the Fe_3O_4 NPs rely on the type of salt utilized (e.g. nitrates, perchlorates, chlorides, sulfates, etc.), ionic strength of the media, the pH value, the growth temperature, the ferric and ferrous ions ratio, and the other factors (e.g. dropping speed of basic solution, stirring rate) [46-48]. Figure 6 reveals a schematic representation of Fe_3O_4 NPs formation during chemical co-precipitation. S. ISLAM et al. [47] investigated the comparative formation of Fe_3O_4 NPs by co-precipitation and hydrothermal methods. The results reveal that in respect of particle size, saturation values of magnetization, and heat dissipation capability, the co-precipitation method is better, whereas in respect of absorbance (reflectance), and particle shape the hydrothermal method is better.

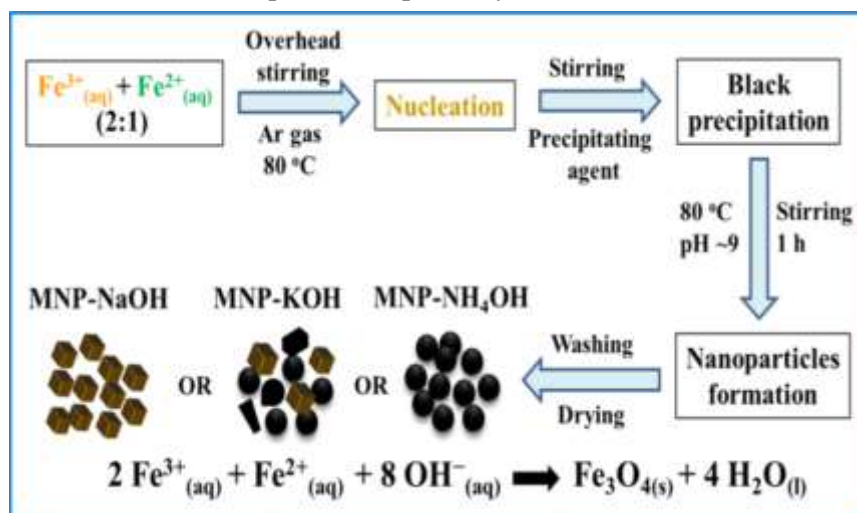


Figure 6: Schematic representation of SPIONs formation during chemical co-precipitation [49].

M. Tajabadi and M. E. Khosroshahi [50], reported the influence of alkaline medium temperature and concentration on significant properties of Fe_3O_4 NPs. ferrous chloride hexahydrate and ferric sulfate heptahydrate are used as iron sources. At two different temperatures i.e. 25 and 70°C, NH_4OH with (0.9-2.1) M concentration was utilized as an alkaline precursor. These results display that the particles prepared at higher temperature (70°C) and minimum alkaline concentration (0.9 M) possess the largest saturation magnetization, at 70°C around 68 emu/gr, in comparison with the smallest particle size at 25°C about 63 emu/gr. R. Rahmawati et al. [51] studied the influence of the frequency of ultrasonic waves and the stirring rate on the particle size of magnetite NPs prepared via co-precipitation protocol. Until 700 rpm, the average crystallite size of Fe_3O_4 NPs reduced from 24.0 to 22.3 nm, then increment to 25 nm up to 900 rpm. T.Q. Bui et al. [52], prepared monodisperse magnetite nanoparticles

by an ultrasonically enhanced co-precipitation process. The TEM images revealed that the magnetite had homogeneously spherical nanoparticles in the form of nanoparticle agglomerates with an average diameter of 10 nm as revealed in Figure 7. Their research indicated that the magnetic responsiveness of Fe_3O_4 NPs generated by co-precipitation is dependent on the particle sizes and that the magnetic responsiveness increment as the particle size decreases.

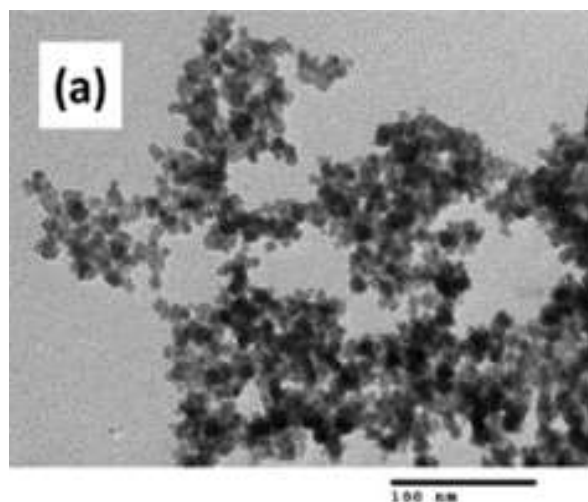


Figure 7: TEM image of Fe_3O_4 NPs [52].

4.3. Sol-gel

Because of its inexpensive cost, low sintering temperature, and capability to modify the size of the particle with homogeneous components, the sol-gel technique is popular [53-58]. The Sol-gel process starts with hydrolysis and poly-condensation to make a gel. Figure 8 showed a schematic diagram of the sol-gel process for the production of nanopowders. This process is an appropriate wet chemical technique for producing metal oxide nanoscale with particular characteristics [27, 30, 59]. One of the most significant disadvantages of this approach is accumulation during the washing operation, which makes it incapable of producing monodispersed nanoparticles [60]. Many researchers successfully prepared monodispersed and non-agglomerated nanoparticles utilizing this strategy to overcome this drawback [27, 61- 68]. Hydrolysis and condensation rates are important parameters that influence the characteristics of final particles. Slower and more controlled hydrolysis generate smaller particle sizes and more distinct characteristics. The solvent should be removed after the solution has condensed into a gel. Higher calcination temperatures are typically required to decompose the organic precursor. The size of the sol particles is determined by the composition of the solution, pH, and temperature [30, 54]. P. Hu et al. [63], synthesized monodisperse Fe_3O_4 NPs with 3-20 nm size via an explosion-assisted sol-gel method. The products were well-crystallized, highly pure Fe_3O_4 NPs according to the XRD and XPS. The influence of various temperatures of (5, 128, and 300) K on the way magnetic behaves was thoroughly investigated. Their finding displayed weakened hysteresis behavior at the temperature increment. At (the Verwey transition) temperature T_v , saturation magnetization (M_s) of 86.2emu/g is the highest. coercivity (H_c) decreases with temperature, while Initial susceptibility (χ_i) increases. O.M. Lemine et al. [64], synthesized Fe_3O_4 particles with an average size of 8 nm and well crystallinity have been prepared via adjusted sol-gel method under supercritical conditions of ethyl alcohol (EtOH). XRD and Mössbauer analysis indicate that the NPs are single phases. The presence of spherical NPs with homogeneous size distribution is revealed by TEM analysis as displayed in Figure 9. At room temperature, SQUID measurements confirm the nanoparticles' ferromagnetic behavior, with a saturated magnetization of 47 emu/g. S. Shaker et al. [54], studied the influence of different annealing temperatures of 200, 300, and 400°C on the particle size. These results reveal that the size of magnetite NPs can change by varying the annealing temperature.



Figure 8: Schematic diagram of sol gel process for production nanopowders [65].

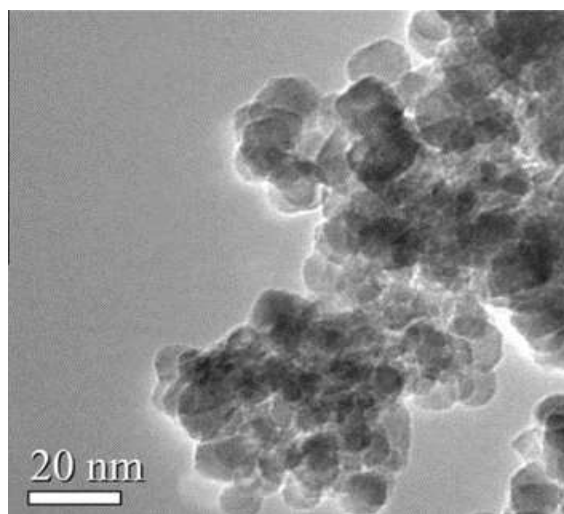


Figure 9: TEM images of the NPs [64].

4.4. Thermal Decomposition

One of the most widely utilized methods for producing monodisperse and highly crystalline IONPs is thermal decomposition [27]. Thermolysis, or thermal decomposition, is the process of treating a substance with heat. The thermal decomposition temperature is the temperature at which the chemical decomposes. This is an endothermic reaction because it requires heat to break the chemical bonds [66]. Figure 10 displays a schematic diagram of magnetite nanoparticles synthesis by thermal decomposition. With the technique, iron oxide NPs have been prepared to utilize the decomposition of organometallic precursors i.e. Fe (cup)₃ (cup = N-nitrosophenylhydroxylamine), Fe(acac)₃ (acac = acetylacetonate), or Fe(CO)₅ (co= carbonyls), after that, oxidation can result in monodispersed aloft -quality iron oxide NPs, although this normally necessitates higher temperatures and a more difficult procedure [7,67]. D. Maity et al. [68], reported a fabricated of water-dissoluble Fe₃O₄ NPs via thermal decomposition of iron (III) acetylacetonate, Fe(acac)₃ in tri(ethyleneglycol). TEM points out that Fe₃O₄ NPs are relatively monodispersing with an average crystallite of 10.7 nm as revealed in Figure 11. The Size and the composition of the product particles are relayed on factors such as the temperature, the surfactant molecule length, and the reaction time [69]. N. J. Orsini et al. [70], have reported succeeding in preparing Fe₃O₄ NPs with diameters d , $7\text{nm} \leq d \leq 12\text{nm}$, by thermal decomposition of Fe(acac)₃. The structural and magnetic characteristics of nanocrystals were studied in relation to different reaction conditions. The most essential parameter controlling the final particle size prepared by thermal decomposition is the heating rate. Table 2 displayed different methods and morphology of magnetite of Fe₃O₄ nanoparticles.

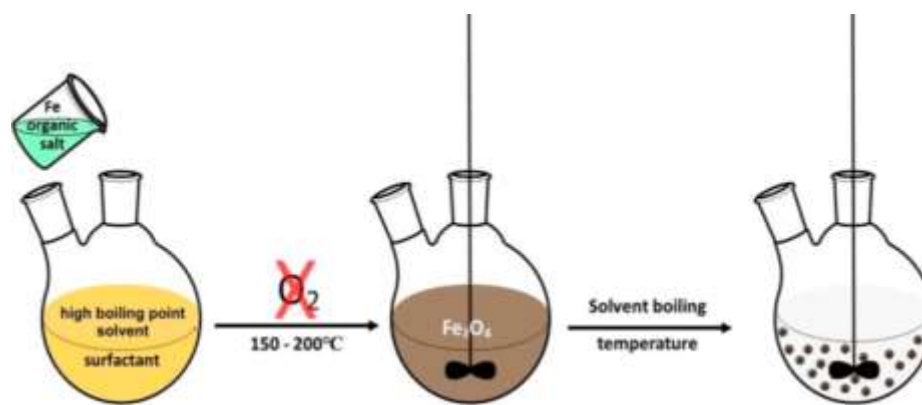


Figure 10: Synthesis of MNPs by thermal decomposition [71].

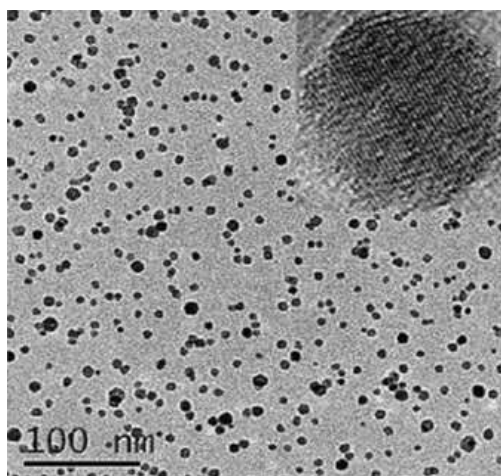


Figure 11: TEM image of the Fe_3O_4 NPs. Inset is the HRTEM image of individual Fe_3O_4 nanocrystals [68].

Table 2: Different methods and morphology of magnetite of Fe_3O_4 nanoparticles.

Method of Synthesis	Precursor	Morphology	Ref.
Hydrothermal	$\text{Fe}_3\text{O}(\text{OCOCH}_3)_6\text{NO}_3$, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$.	Spherical NPs with an average diameter of 10 nm, were prepared at 180°C for 20 h and the value of pH is 8.6.	[72]
Hydrothermal	$\text{Fe}_3\text{O}(\text{OCOCH}_3)_6\text{NO}_3$, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$.	Nanorods with an average width of about 25nm and a length of about 200nm.	[72]
Co-precipitation	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$	Nanoparticles were nearly spherical and non-aggregated with a mean size of 10 nm.	[73]
thermal decomposition	iron acetylacetonate (acac) iron oleate complexes	Uniform nanoparticles are composed of a mixture of triangular, cubic, and diamond-shaped particles, with an average particle size of 11 nm.	[74]

thermal decomposition	Fe (NO ₃) ₃ ·9H ₂ O	Ultrafine particles which are closely packed form nano-aggregates (≤ 100 nm). There is a powerful agglomeration of NPs with the size of 10 nm.	[75]
sol–gel	Fe (NO ₃) ₃ ·9H ₂ O	Nanoparticles had been agglomerated, particles of grain dimensions having a range of 15–30 nm.	[76]
Solvothermal	FeCl ₃ · 6H ₂ O	A spherical shape of Fe ₃ O ₄ particles has uniform sizes and good dispersibility with a mean diameter of 326 nm.	[77]

5. Conclusions

Nowadays, Magnetic NPs have piqued the attention of researchers due to their intriguing properties and diverse applications. Many chemical synthesis routes, including sol-gel, thermal decomposition, hydrothermal, and co-precipitation, have revealed some benefits and disadvantages for the preparation of nanoparticles. Magnetite nanoparticles' (sizes and geometries) and magnetic properties are two important properties that can be obtained using suitable synthetic approaches. As a result, the sizes and shapes of magnetic Fe₃O₄ NPs are critical structural factors that influence many characteristics of NPs and their capabilities in various applications.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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