

Chapter

Magnetite Nanoparticles (Fe_3O_4) for Radio-Frequency and Microwave Applications

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Abstract

The size and shape dependent tunable electromagnetic (EM) properties of magnetite – Fe_3O_4 nanoparticles makes them an attractive material for various future electronics and biomedical device applications such as tunable attenuators, miniaturized isolators and circulators, RF antennas, EM shielding, and biomedical implants etc. The strategic design of RF devices requires specific dielectric and magnetic properties according to the applications, which in turn depends on the size and shape of the particles. At nanoscale, iron oxide's magnetic and dielectric properties are very different from its bulk properties and can be tuned and enhanced by utilizing different synthesis approaches. In this chapter, we summarize electromagnetic properties of magnetite (Fe_3O_4) nanomaterials such as, complex permeability, complex permittivity, magnetic and dielectric loss tangents, saturation magnetization, temperature dependence, and ferromagnetic resonance; and how these properties can be optimized by varying different synthesis parameters. Finally, Fe_3O_4 nanocomposites will be explored by using different synthesis approaches for implementation of RF and microwave applications and we will conclude the chapter with future recommendations.

Keywords: Fe_3O_4 nanoparticles, morphology, magneto-dielectric properties, RF and microwave region, magnetic loss tangent

1. Introduction

Over the past few decades, nanotechnology has expanded its applications exponentially in all aspects of life ranging from biomedical, chemical, material engineering to integrated electronics [1–5]. In nanotechnology, functional nanoparticles with size ranging from 1 to 100 nm have been widely studied [6]. The unique and specifically tailored structure and size dependent properties of the nanoparticles make them extensively important for research and development for various applications such as environment, healthcare, medical, defense, electronics, and so on [7–9]. Nanoparticles have different properties from their bulk counterparts because as the size of the particle decreases, surface effects (more atoms are exposed at the surface of particle, thus leading to highly sensitive and reactive surfaces) and other atomic effects such as quantum confinement effect in

electronic structure comes into play [10, 11]. The key to achieve novel chemical, structural, magnetic, physical and mechanical properties of nanoparticles is the large surface to volume ratio [12].

Recently metal oxide nanoparticles such as iron oxide has garnered considerable attention due to its unique structural, electrical, and magnetic properties which, have numerous applications in areas such as data storage, memory devices, water purification, bioprocessing, drug delivery, hyperthermia, magnetic resonance imaging (MRI), biosensors, electronic devices, aerospace applications, etc. [13–16]. Iron oxide is a compound, which can be found in nature in different phases. The most common ones are hematite (α - Fe_2O_3), magnetite (Fe_3O_4) and maghemite (γ - Fe_2O_3) [17, 18]. All of these forms show promising properties such as biocompatibility and relatively low toxicity, high stability under the presence of magnetic field, superparamagnetic response when the particle sizes are kept below 50 nm, and ease of synthesis process and surface treatment [19]. Among them, the magnetite (Fe_3O_4) has been widely used in most practical applications, due to the coexistence of ferrous and ferric cations in Fe_3O_4 , which results in fascinating magnetic and structural properties (e.g. high magnetic moment due to valence d electrons) [20]. The high demand and superb performance of magnetite – Fe_3O_4 , is attributed to their microstructure, particle size, more active electronic sites, and high surface area to volume ratio [21–24]. The performance of magnetite (Fe_3O_4) nanoparticle properties strongly depends on the oxide phase, the particle morphology, the shape and size distribution, the internal composition (e.g., impurities, grain boundaries and the surface chemistry). Therefore, for a particular application, synthesis methods and procedures must be tailored and optimized [22]. Synthesis methods dictate the crystalline properties, size, shape, and quality of the magnetite nanoparticles. Hence, it greatly affects the magnetic, dielectric and loss properties. Meanwhile, sizes and shapes are also critical; shape change shows crystal facets, and the atomic arrangements in each facet have reflective effects on its electronic properties. There is a growing demand of novel magnetic materials in electronic industry. Here, we will overview and briefly discuss the synthesis methods while focusing on radio frequency (RF) and microwave electronic applications of magnetite (Fe_3O_4) nanoparticles.

Magnetite (Fe_3O_4) nanoparticles can be synthesized using different methods such as physical (laser ablation arc discharge, combustion, electrodeposition, and pyrolysis), chemical (sol–gel synthesis, microemulsion, hydrothermal, coprecipitation, Polyols, thermal decomposition) and biological methods (Protein mediated, plant mediated, bacteria mediated, fungi mediated). Different shapes and sizes of Fe_3O_4 (nanorod, porous nanospheres, nanocubes, distorted cubes, core shell and self-oriented flowers) can be synthesized using same synthesis procedures, by using the optimum synthesis parameters like particular precursor of iron salts, pH levels, and temperature variations etc. [25, 26]. These synthesis methods are easy to implement while playing a major role in controlling the morphology and electromagnetic properties of Fe_3O_4 nanoparticles. In order to make Fe_3O_4 nanoparticles compatible with different applications, proper functionalization and surface modification of Fe_3O_4 is very important [27, 28]. Surface modification of the Fe_3O_4 nanoparticles using different stabilizing agents (PVP, oleic acid, sodium oleate etc.) is a necessary step after or during the synthesis process to make them both biocompatible and stable [29, 30].

For RF and microwave electronics, tunable or reconfigurable devices are becoming important to cause a growing interest of enabling nanotechnology in new wireless devices [31]. Magnetic materials have been used effectively for tunable and reconfigurable of components such as inductors, antennas, and phase shifters [32, 33]. By using

tunable properties of Fe₃O₄ nanoparticles in these devices, one can control not only their frequency response but also helpful in improvement of electromagnetic behavior of these devices at a particular frequency [34, 35]. In this chapter, we will discuss the synthesis procedures of magnetite (Fe₃O₄) nanoparticles and their usage in RF and microwave applications. The development of sustainable synthesis approaches for these nanoparticles and investigations of how the structural properties including shape and size of magnetite nanoparticles can enable the tuning of electromagnetic properties for different device applications will be presented.

2. Synthesis methods

As mentioned above, there are different approaches to synthesize magnetite (Fe₃O₄) nanoparticles, which includes physical, chemical, and biological methods. The properties of Fe₃O₄ nanoparticles determine its field of applications. The most widely used synthesis approaches are chemical co-precipitation, thermal decomposition, hydrothermal method, Polyols method and microemulsion method [25].

As shown in the figure, chemical methods are mostly widely used as they are cost effective and easy to handle. Some of the most common synthesis methods are summarized below [25].

2.1 Co-precipitation methods

Co-precipitation synthesis is the most common technique for the synthesis of magnetic magnetite (Fe₃O₄) nanoparticles because of its low cost, environment friendly precursors and simple experimental procedure that occurs at moderately low temperature (20°C - 90°C) [6]. This method is popular because of water based precursor solutions, where simultaneous precipitation of ferrous and ferric ions can occur due to the addition of base in the solution while sustaining a constant pH level. Fe (II) and Fe (III) salts are used in different basic aqueous solutions such as NaOH and NH₄OH to form magnetite (Fe₃O₄) nanoparticles. Nanoparticle size between 5 nm and 20 nm range can be synthesized using this method [11]. Experimental conditions such as Fe²⁺ and Fe³⁺ salt chlorides, sulphates, nitrates, ratio of Fe²⁺ and Fe³⁺ ions in the solution, ionic strength of the solution, pH value of the solution and reaction temperature are very critical parameters to achieve desired size, shape, microstructure, and magnetic properties. Key literature findings about the effects of some of these conditions on nanoparticles properties with a special focus on electronic properties will be detailed below. **Figure 1** shows the typical co-precipitation technique experimental set-up using multistage flow reactor for continuous synthesis of Fe₃O₄ nanoparticles [36].

It is known that co-precipitation method typically results in low saturation magnetization and broad particle size range due to variation in magnetite (Fe₃O₄) nanoparticles core size and agglomeration, which are the main drawbacks [37, 38]. In order to reduce agglomeration and oxidation of Fe₃O₄ nanoparticles, different surface acting reagents and functional materials such as polyethylene glycol (PEG), Polyvinyl Alcohol (PVA), dextrin, Polyvinylpyrrolidone (PVP) etc. can be added during the reaction [39–42].

Radon *et al.* in 2017 studied effect of different organic modifiers (glycol, PVP, citrate and dextrin) on the structural and optical properties of Fe₃O₄ nanoparticles [43]. It was observed that organic modifiers with an exception of glycol facilitate

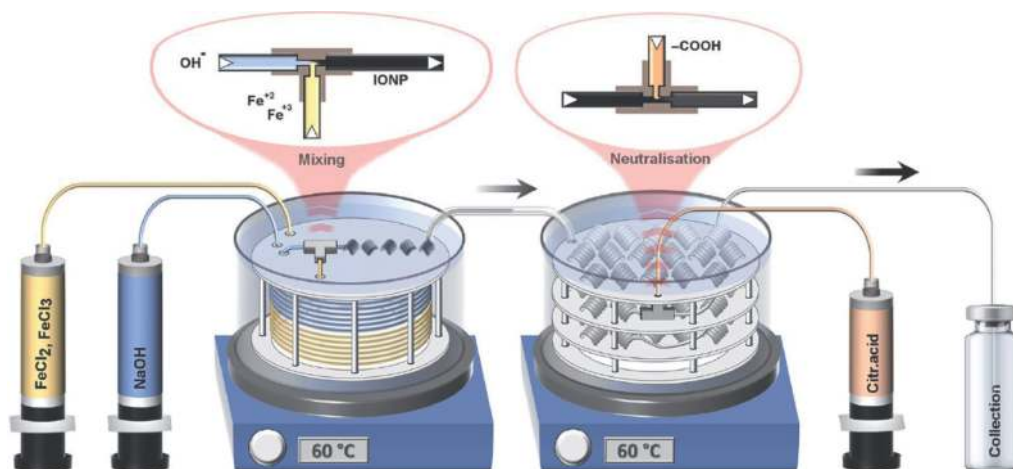


Figure 1. Co-precipitation method for the synthesis of Fe_3O_4 nanoparticles using multistage flow reactor [36].

reduction of particle size. Particle size range of 2.9 nm to 12.9 nm was reported by modifying co-precipitation process. Fe_3O_4 nanoparticles synthesized with tartaric acid in the solution have the smallest mean particle size along with largest band gap energy [43]. The results showed that an increase in particle size leads to wider optical bandgaps. **Figure 2** shows schematic representation of synthesized Fe_3O_4 nanoparticles along with organic modifiers using XRD, TEM and FTIR data [43].

Similarly, Anbarasu *et al.* studied the effect of PEG on the crystallite size of Fe_3O_4 nanoparticles and saturation magnetization. With an increase in weight of PEG coating (1 g - 3 g) on Fe_3O_4 nanoparticles, crystallite size decreases from 14.96 nm to 10.85 nm, while saturation magnetization also decreases from 62 emu/g to 51 emu/g, accordingly [44].

Saragi *et al.* in 2018 studied the effect of reaction temperature variation on the structure and size of the Fe_3O_4 nanoparticles. They reported an increase in mean particle size from 10.14 nm to 11.66 nm with an increase in reaction temperature from 25 to 80°C [45]. It was reported that smaller crystallite size was measured for low temperature synthesis. It was observed that band gap energy for Fe_3O_4 nanoparticles has decreased from 1.76 eV to 1.14 eV with an increase in particle size. **Figure 3** shows the variation of complex permittivity spectra and magnetization of Fe_3O_4 nanoparticles with respect to temperature variation [46]. There was a nonlinear relation between complex permittivity and temperature. Similar effects of temperature variation on dielectric and structural properties of Fe_3O_4 nanoparticles have been reported by Radon *et al.* in 2018 [46]. The permittivity and dielectric loss as seen in **Figure 3** exhibited a nonlinear behavior in response to temperature variation [45]. It is observed the dielectric property was mostly dominated by polarization process. The electromagnetic shielding mechanism of nanocomposites can be attributed to absorption process of low-reflection electromagnetic shielding composites.

Optimization of co-precipitation synthesis parameters in order to control the particle size and polydispersity can be quite challenging, extensive ongoing research have been carried out to understand the mechanism of particle formation so that particle structures/properties can be tailored for applications.

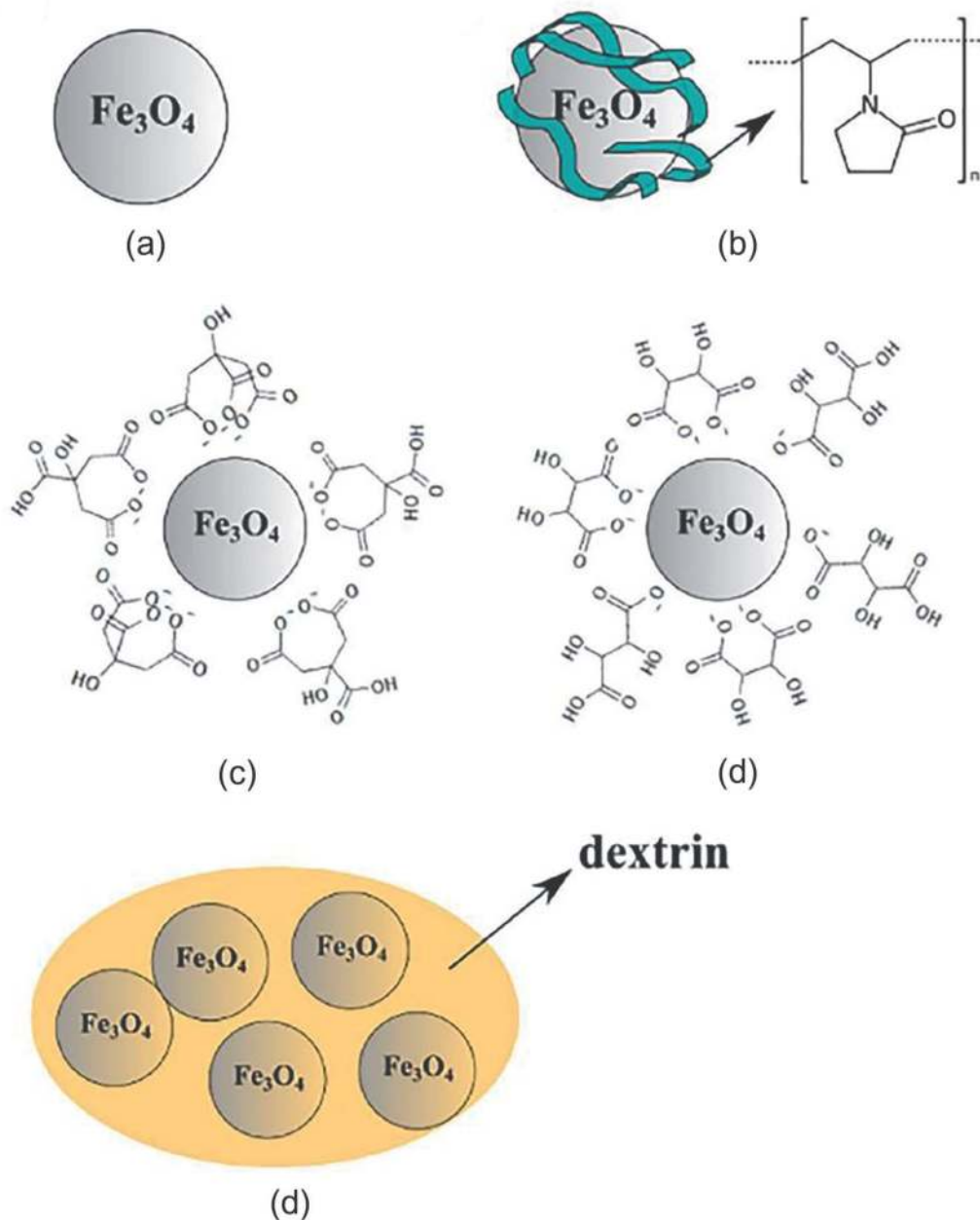


Figure 2. A schematic representation of synthesized Fe_3O_4 nanoparticles along with organic modifiers using XRD, TEM and FTIR data by showing (a) Fe_3O_4 and glycol; (b) Fe_3O_4 and PEG; (c) Fe_3O_4 and citrate; (d) Fe_3O_4 and tartrate; and (e) Fe_3O_4 and dextrin [43].

2.2 Thermal decomposition

Thermal decomposition is a synthesis of Fe_3O_4 nanoparticles using decomposition of iron precursor at high temperature in organic phase solution [47]. In this method, precursors of iron (III) acetylacetonate, $\text{Fe}(\text{acac})_3$, iron nitro sophenylhydroxylamine or iron pentacarbonyl are used in oleic acid or lauric acid, which are oxidized at high

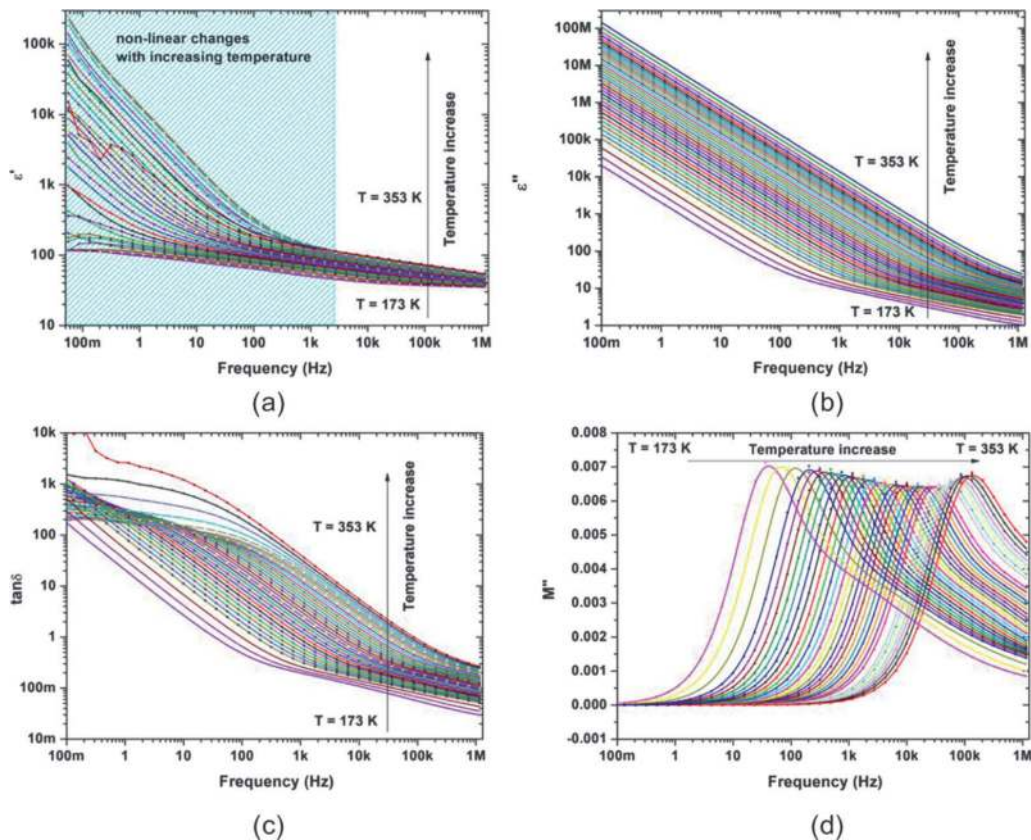


Figure 3.

Variation of complex permittivity spectra and magnetization of Fe_3O_4 nanoparticles with respect to temperature variation [46].

temperature to make monodisperse Fe_3O_4 nanoparticles [6]. **Figure 4** presents a conceptual illustration of experimental process to synthesis of monodisperse Fe_3O_4 nanoparticles [47].

The thermal decomposition method can be used to synthesize monodisperse nanoparticles of up to 20 nm in size with a tight size distribution. Wetterskog *et al.* in 2015 have synthesized nanospheres and nanocubes shaped Fe_3O_4 nanoparticles using thermal decomposition method [48]. A size distribution between 5 nm and 27 nm was reported for both nanocubes and nanospheres. Their work demonstrated that size of nanoparticles can be tuned by reaction temperature and shape can also be tuned by addition of oleic acid or sodium oleate during the synthesis. **Figure 5** shows variation of shape from nanocube to nanosphere with the addition of sodium oleate [48]. Similar effects of oleic acid or sodium oleate on the shape of Fe_3O_4 nanoparticles have been reported in the literature [49–52]. Li *et al.* in 2010 fabricated oleic acid coated Fe_3O_4 nanoparticles heated at 320°C under nitrogen atmosphere with uniform shapes and sizes [53]. To address the stabilization issue, different stabilizing agents such as bilayer oleic acid, fatty acids, phenol etc. were studied in the past [47, 54, 55]. For example, Wang *et al.* in 2012 used phenol as a reducing agent and stabilizer for the formation of stable water soluble Fe_3O_4 nanoparticles [56]. The main disadvantage of this method is it requires nanoparticles to dissolve in nonpolar solution for storage and cannot be scaled up for industrial production [57]. Using thermal decomposition methods, high saturation magnetization values with low coercive fields can be achieved by using high

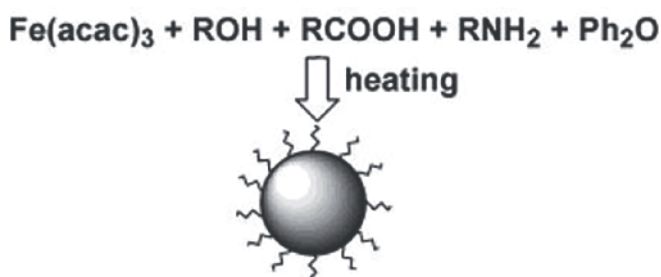


Figure 4. Conceptual illustration of synthesis process of monodisperse Fe_3O_4 nanoparticles using thermal decomposition method [47].

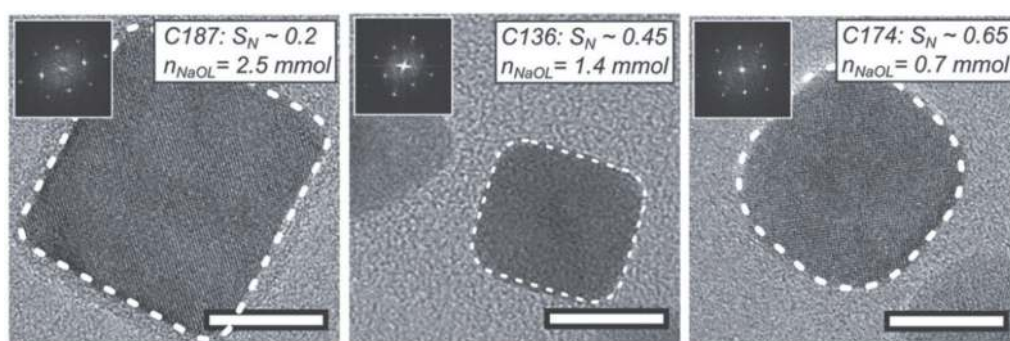


Figure 5. Variation of shape from nanocube to nanosphere vs. adjusted addition of sodium oleate [48].

reaction times in the process. *Vuong et al.* in 2015 showed high values of saturation magnetization up to 70 emu/g with reaction time of 120 mins [58].

2.3 Polyol method

Polyol method is a well-known technique to synthesize defined shape and size-controlled metallic, oxide, and semiconductor nanoparticles such as magnetite (Fe_3O_4) nanoparticles [25]. This method involves chemical reduction of metal salts in polyols such as polyethylene glycol at high temperature. The average size of these nanoparticles can be controlled by reactive mediums and this method is widely used to obtain nanoparticles of size up to 100 nm [21]. The shape, size, particle growth and yield depend upon the type of polyols, salt ratio, concentration, and other physiological condition. Polyol and polyethylene glycol are normally used as solvents, which can dissolve inorganic compounds and offer a wide range of temperature for the reaction. Polyols act as both stabilizer and reducing agent in the reaction and help in prevention of agglomeration and control of particle growth [59]. *Abbas et al.* in 2013 studied the effect of polyethylene glycol as the stabilizer and reducing agent for the synthesis of hydrophilic, monodisperse superparamagnetic Fe_3O_4 nanoparticles for biomedical applications [60]. Similar prior studies have been reported using different polyols such as ethylene glycol, di ethylene glycol, tri ethylene glycol, tetra ethylene glycol and propylene glycol, polyethylene glycol [61–65].

There are also a variety of prior works in the literature, which utilizes solvothermal polyols method to synthesize different Fe_3O_4 cluster sizes for better magnetic

properties such as saturation, magnetization and coercivity. In solvothermal polyol method, Fe_3O_4 clusters can be prepared by change of reaction conditions of the solvothermal process and by utilizing sodium acetate [66]. Leung *et al.* in 2009 synthesize Fe_3O_4 clusters of different size by varying the reaction conditions [67]. They are the first group that studied the effect of solvent composition on Fe_3O_4 cluster size. Similar studies have been reported by different groups on controlling the size of Fe_3O_4 clusters [68], core/shell ($\text{Fe}_3\text{O}_4/\text{ZnO}$) submicron particles [69], PAA modified hydrophilic Fe_3O_4 nanoparticles [70] and Fe_3O_4 nanoparticles [71].

Sayed *et al.* in 2015 utilized microwave assisted solvothermal polyols to synthesize six different shaped Fe_3O_4 nanoparticles using different iron salt as precursors – nanorods, nanohusk, distorted nanocubes, nanocubes, porous spheres and self-oriented flowers [72]. These shapes of Fe_3O_4 were synthesized using KCC-1 synthesis protocol [73], which has involved various iron salts as precursors, cetyltrimethylammonium bromide (CTAB) as a template, along with utilization of cyclohexane-water-pentanol as a reaction solvent and urea as hydrolyzing agent. **Figure 6** shows the SEM images of different shaped of Fe_3O_4 nanoparticles [72].

2.4 Hydrothermal method

Hydrothermal synthesis is the most commonly used method for the preparation of nanomaterials. This is a solution reaction-based approach, which utilizes a wide temperature range from room temperature to high temperatures [74]. To control the morphology of the nanoparticles, low-pressure or high-pressure conditions can be used in the reaction. Pressures above 2000 psi needs to be maintained in hydrothermal synthesis method [25]. The compositions, morphology, particle size of nanomaterials to be synthesized can be well controlled by temperature variation in combination with right precursors in hydrothermal synthesis through liquid phase or multiphase chemical reactions. The particle size and size distribution can also be controlled with precursor concentration [21]. The main drawback of this method is that it needs expensive reactors [1].

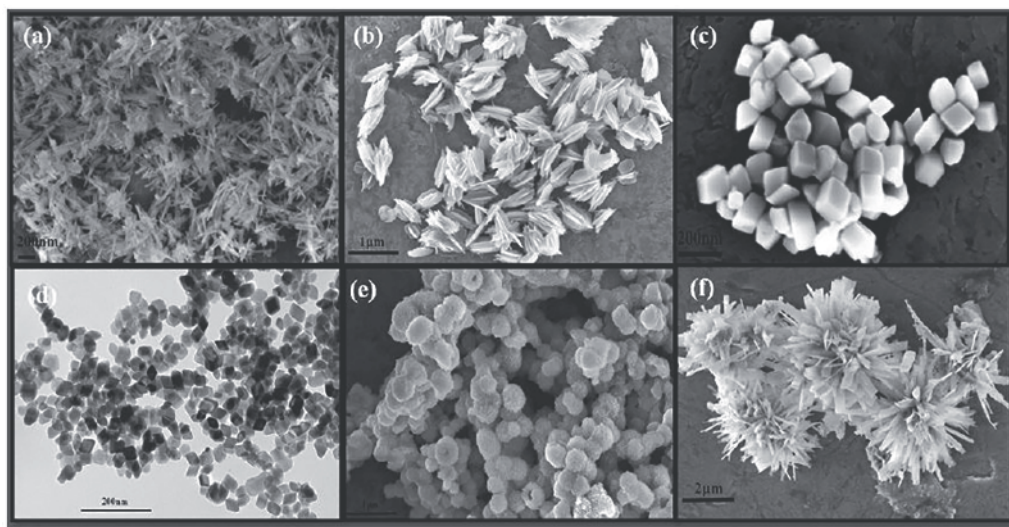


Figure 6. SEM images of six different shaped of Fe_3O_4 nanoparticles obtained by microwave assisted solvothermal polyol method by using KCC-1 synthesis protocol, including: (a) Nanorod, (b) Nanohusk, (c) distorted cubes, (d) Nanocubes, (e) porous spheres, and (f) self-oriented flowers [72].

Gomez *et al.* in 2019 studied the effect of temperature on the morphology of the hydrothermally synthesized Fe_3O_4 nanoparticles. The shape was controlled by the temperature of the reaction; at 120°C, 140°C, and 160°C, to obtain quasi-spheres, octahedrons, and cubes, respectively [75]. **Figure 7** shows the SEM images of Fe_3O_4 nanoparticles obtained at different temperatures [75]. Similar studies were focused on controlling the shape and size of the Fe_3O_4 nanoparticles by controlling temperature, solvent, precursor salt, reducing agent, and so on, while using this hydrothermal method [76–80].

2.5 Microemulsion method

Microemulsion is an isotropic and thermodynamically stable single phase formed by mixing oil, water and surfactants; where oil and water are immiscible, and surfactant has an amphiphilic behavior [81]. There are three main categories of microemulsions - oil in water, water in oil and bi-continuous [1]. Microemulsion method has been known

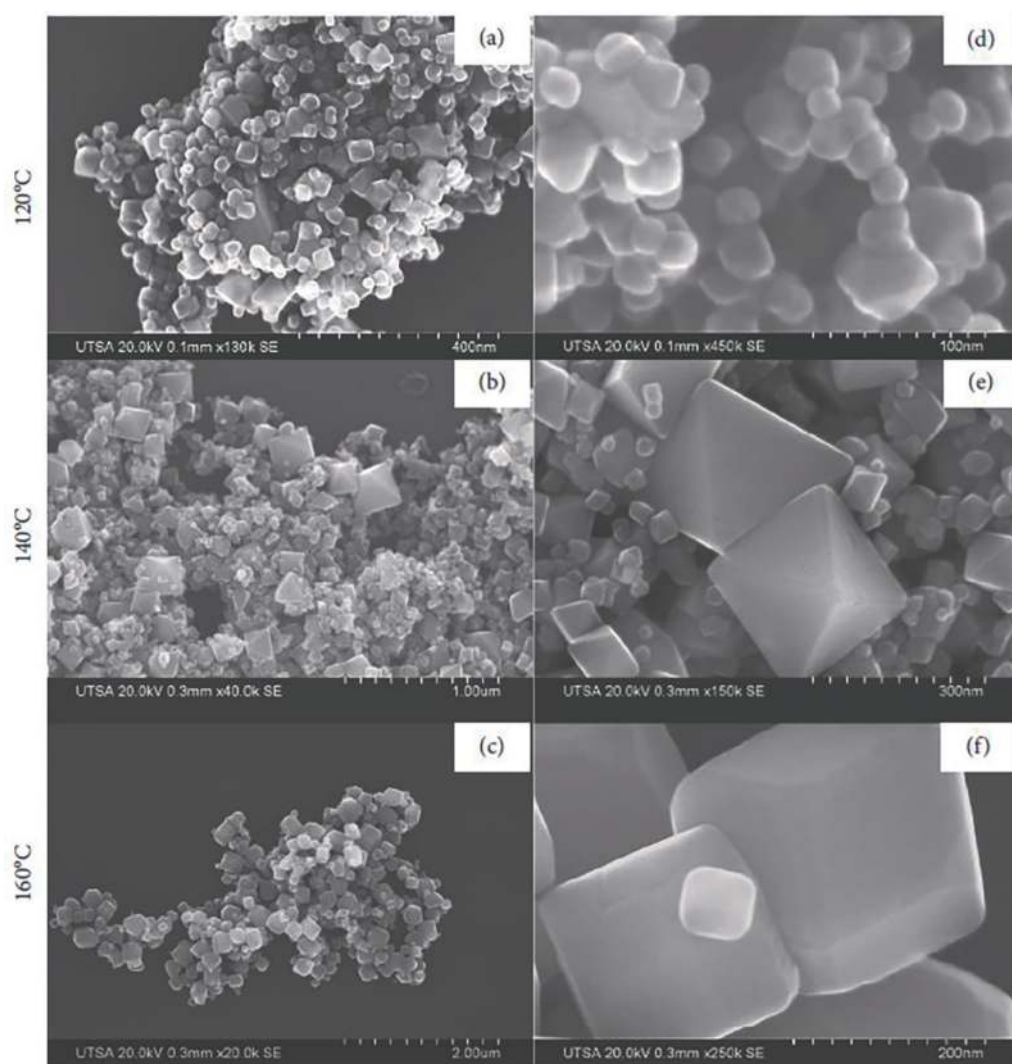


Figure 7. SEM images of Fe_3O_4 nanoparticles synthesized using hydrothermal method at - (a) 120°C, (b) 140°C, (c) 160°C, where (d) to (f) are zoomed-in SEM photos of the nanoparticles at the corresponding temperatures [75].

to produce narrow particle size distribution between 4 and 15 nm with different shapes. Synthesis of Fe₃O₄ nanoparticles with controlled size and shape can be carried out in water-oil microemulsion, which consists of cationic or non-ionic surfactant (Triton-X), a co-surfactant (n-hexanol, glycols, 1-butanol), oil phase (n-heptane, n-octane, cyclohexane) and aqueous phase. Microemulsion can be carried out through addition of aqueous solution with iron precursor to the surfactant mixture [6]. The major drawback of this method is that the scale up of this method from laboratory scale to mass production at industrial levels could be difficult; particle size and shape changes significantly at large scale despite maintaining the same reaction conditions as lab experiments.

Many prior studies have been reported on the controlled synthesis of Fe₃O₄ nanoparticles using microemulsion method [82–85]. In order to increase the stability of Fe₃O₄ nanoparticles and avoid agglomeration, they have been encapsulated

Methods	Size (nm)	Shape	Saturation Magnetization Ms. (emu/g)	Advantages	Disadvantages
Co-precipitation	3–100	Spherical	20–80	Low to mild temperature, high yield, scalable, inexpensive synthesis, simple purification	Agglomeration, polydispersity
Thermal decomposition	3–80	Spherical, 1D and 2D	Less than 90	Narrow size distribution, high crystallinity, size and shape control	Long reaction time, high temperature, organic medium, expensive, low yield
Polyols Method	10–1000	0D,1D,2D,3D	20–120	size and shape control, less agglomeration, high yield,	Broad particle size distribution
Hydrothermal	2–1000	0D,1D,2D,3D	20–110	High purity nanoparticles, medium temperature, low cost, use stabilizers in reaction to control agglomeration, high yield, aqueous reaction medium	Long reaction time, broad particle size distribution
Microemulsion	4–50	Spherical and cubic	30–110	Low temperature, ambient atmosphere, narrow size distribution, controllable size	Long reaction times, agglomeration, low yield, difficult to remove surfactants

Table 1. Comparison between different synthesis methods of magnetite (Fe₃O₄) nanoparticles.

with silica precursor, which significantly increase the stability of nanoparticles and protecting them from oxidation [79, 86, 87]. Asab *et al.* in 2020, reported silica coated Fe₃O₄ nanoparticles using water-in-oil microemulsion method for the application of antimicrobial activity. It was observed that silica-coated Fe₃O₄ nanoparticles exhibited homogeneous distribution of particles with relatively less severe agglomerate of the particles [86]. Comparison among different synthesis methods is presented in **Table 1**.

3. Application of magnetite (Fe₃O₄) nanoparticles

Magnetite (Fe₃O₄) nanoparticles are well suited for a wide variety of scientific and engineering applications in numerous fields, due to their strong superparamagnetic and surface properties. Detailed application areas are summarized in **Table 2**. We herein specifically focus on radio frequency (RF) and microwave applications.

Area	Applications
Biomedical and healthcare	Drug delivery [88–90], magnetic hyperthermia [91–94], MRI imaging [42, 95, 96], magnetic separation, controlled drug release, cellular therapy, cell separation and handling of cells [97, 98], purifying cell populations, diseases of the musculoskeletal system, severe inflammation, toxicity [99]
Agriculture	Nano fertilizers, nano fungicides, nano pesticides [100, 101]
Environment	Wastewater treatment, catalyst coatings [102–104]
Recording and storage	Ferrofluids, external magnets [105]
Industries	Catalyst [106, 107]
Textile	Nanofibers, sensors, smart materials [108–110]
Defense	Sensors, nanocomposites, smart materials [111, 112]
Electronics	Printed electronics, spintronics and quantum dots [113, 114]

Table 2.
Scientific and engineering fields of applications for magnetite (Fe₃O₄) nanoparticles.

4. Application of magnetite (Fe₃O₄) nanoparticles for RF and microwave region

With the continuous technological advancements and emerging applications in biomedical devices and electronics in RF and microwave regions, the strategic design of suitable electromagnetic materials requires controlled and well-tailored dielectric, magnetic and loss properties. There is a growing demand to increase the operating frequency of RF and microwave devices. Magnetite (Fe₃O₄) nanoparticles have recently shown great promises for these applications due to their exciting and superior magnetic properties at high operating frequencies [35]. Nevertheless, as an emerging research area with an aim to employ Fe₃O₄ nanoparticles for unique RF applications, there are relatively limited prior works at this stage.

Fe₃O₄ nanomaterial is the among the very few magnetic materials that exhibits excellent tunable properties using different synthesis approaches. Fe₃O₄ nanoparticles have attracted considerable attentions because of its shape and size tunability, which in turn impact the magnetic and loss properties. The tunable electromagnetic properties of Fe₃O₄ nanoparticles are uniquely suited for designing RF/microwave devices

due to their structural and size dependent magnetic and dielectric properties, which can further tuned by external magnetic fields [115, 116]. Meanwhile, self-biased soft magnetic ferrites have been recently explored to exhibit unique properties by exploiting the anisotropy of magnetic material [117–119]. Fe_3O_4 nanoparticles polymer composites have exhibited unique attributes for biomedical device and electronic applications, which require tuned, light weight, robust, flexible and cost-effective devices such as antennas [120].

4.1 Tunable electromagnetic properties of magnetite (Fe_3O_4) nanoparticles for RF and microwave devices

In 2008, Kuanr *et al.* studied the size dependent magnetic properties of Fe_3O_4 nanoparticles for microwave devices [121]. To study the electromagnetic properties of Fe_3O_4 nanoparticles, nanoparticles fluid with oleic acid was spin-coated on a GaAs substrate equipped with a Cu coplanar waveguide (CPW). **Figure 8** shows the transmission response and frequency shift with varied Fe_3O_4 nanoparticle sizes. They reported an increase in resonance frequency with an increase in particle size from 4 nm to 14 nm and then starts to decrease as shown in **Figure 8** (c) [121]. Change in magnetization from $M_s = 0.14, 0.19, 0.25,$ and 0.29 kG for the 4, 6, 8, and 10 nm nanoparticle samples, respectively, was reported. These values are consistent with other reported prior works. The Fe_3O_4 nanoparticles used were synthesized by thermal decomposition method.

Recently, Jadav *et al.* in 2020 studied the effect of particle size on the microwave behavior of Fe_3O_4 nanoparticles in RF region (250 MHz–3 GHz) [122]. It was observed that maximum loss tangent increases, and minimum reflection loss (RL) decreases with a increment of mean particle size. For instance, the maximum magnetic loss was increased by 55.6% and minimum RL was decreased by 34.5% by increasing the mean size of Fe_3O_4 nanoparticles from 11 nm to 16 nm. **Figure 9** shows the magnetic loss tangent and return loss variation with frequency for 4 different nanoparticle sizes (MF1–10 nm to MF4–17 nm) in magnetic fluid [122]. The maximum loss tangent and minimum RL was achieved for 17 nm particle size (MF4) because of high magnetic permeability resulted from a large mean particle size. These properties of Fe_3O_4 nanoparticle are affected by an external magnetic field strength between 0 Oe and 380 Oe, while retaining particle size. For example, resonance frequency shifted form 1.28 GHz (0 Oe) to 2.03 GHz (368 Oe) by applying a 368 Oe magnetic field, accompanied by an increase in magnetic loss tangent for a 10 nm particle sized sample. This was attributed to the fact that shape and size distribution of nanoparticles leads to change in anisotropy constant, which in turn affect the magnetic permeability and magnetic losses. As the magnetic field strength increases, resonance frequency shifts to higher value. Also, the bandwidth increases for largest particle size. The magnetic loss tangent decreases with an increase in magnetic field strength for the largest particle size, while the field strength in the nanoparticle fluid reaches to the maximum and then drop to low value for small size particle. The Fe_3O_4 nanoparticles studied in this work were synthesized using co-precipitation method. Different pH variations were used to synthesize different size nanoparticles at constant temperature for the measurement of these microwave properties [122].

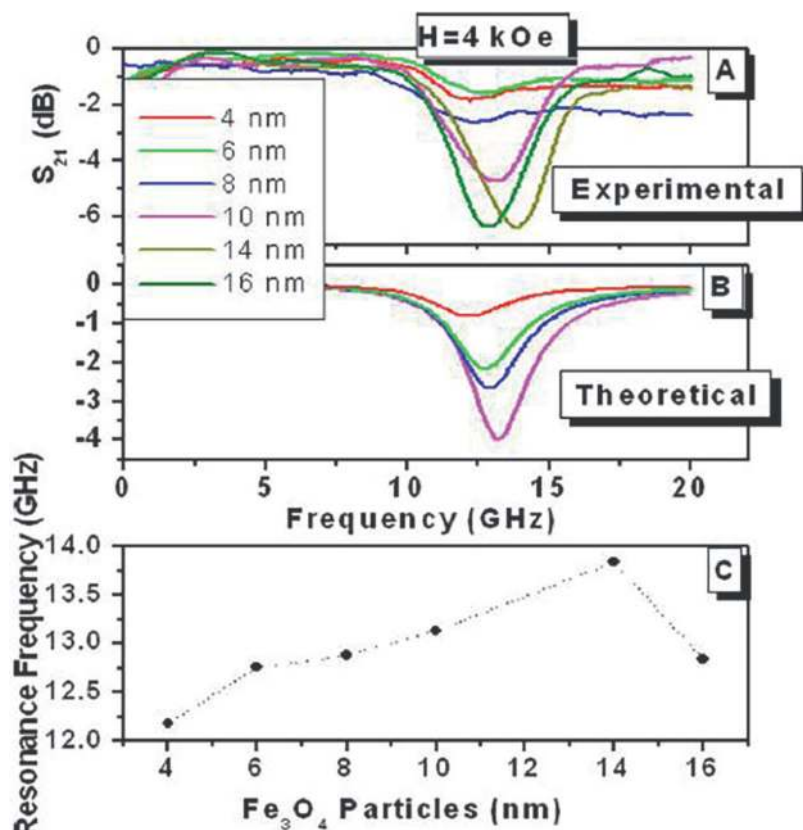


Figure 8. (a) Measured transmission responses vs. particle sizes under a 4 kOe of external magnetic field; (b) theoretical model-predicted transmission responses vs. particle sizes under a 4 kOe of external magnetic field; and (c) measured resonance frequency vs. Fe_3O_4 particle size [121].

Similarly, the effect of particle concentration, external magnetic field, frequency dependence of RF and microwave properties [35, 123, 124], agglomeration effects on the effective electromagnetic properties of composites with magnetic Fe_3O_4 nanoparticles [125, 126] have been studied and reported in the literature. For example, Li et al. in 2015 reported water soluble Fe_3O_4 nanoparticles coated using surface double-layered self-assembly method. The sodium alpha-olefin sulfonate (AOS) was used as the coating material for better superparamagnetic properties [127]. It was confirmed that AOS double coated Fe_3O_4 magnetic nanoparticles showed less agglomeration as compared to Fe_3O_4 nanoparticles. Saturation magnetization value of about 44.45 emu/g and the blocking temperature T_B 170 K were reported for Fe_3O_4 -AOS capped nanoparticles which are ideal values for biomedical applications.

Fabrication of heterostructures is another way to tailor the magnetic properties of the soft magnetic ferrites such as the ones based on Fe_3O_4 nanoparticles for planar device applications (e.g., inductors and patch antennas) including multi-layer ferrite materials with isostructural and non-isostructural materials, (e.g., $\text{Fe}_3\text{O}_4/\text{NiO}$, $\text{Fe}_3\text{O}_4/\text{CoO}$, $(\text{Mn}, \text{Zn})\text{Fe}_2\text{O}_4/\text{CoFe}_2\text{O}_4$, etc.). The combination of Fe_3O_4 soft magnetic ferrite layer and a piezoelectric layer can lead to new and exciting RF and microwave applications such as antenna, sensors etc. [20].

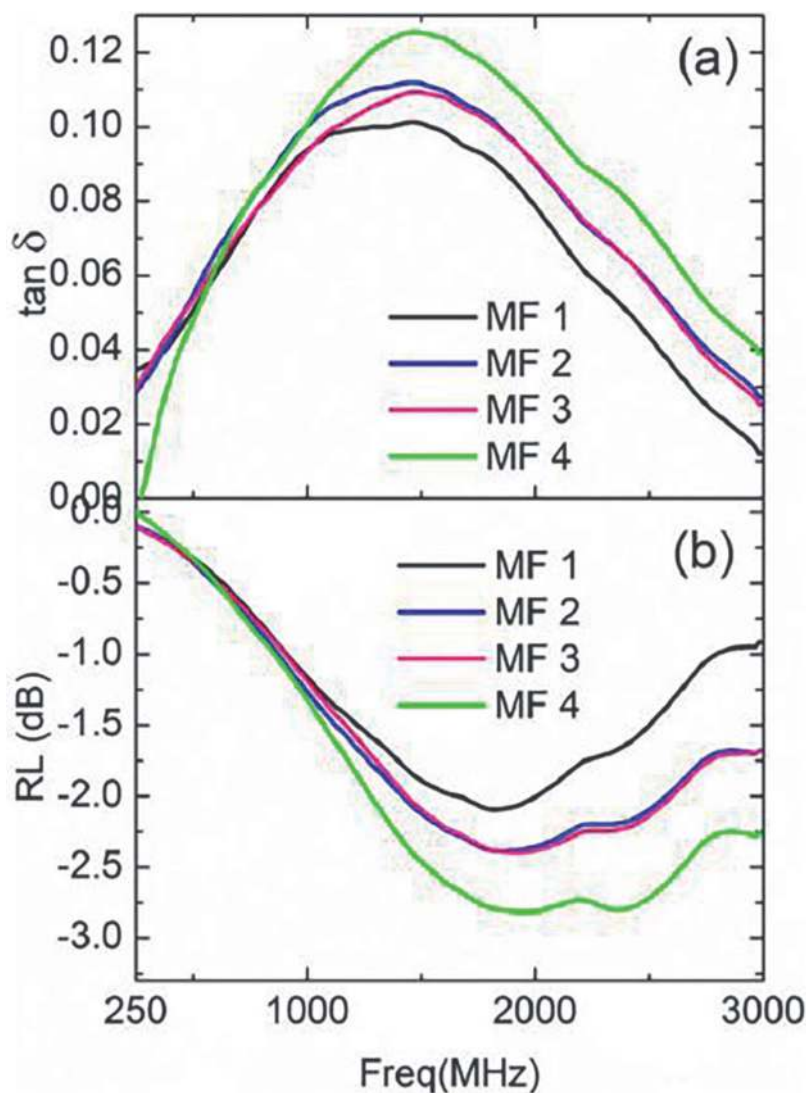


Figure 9.

(a) Variation of magnetic loss tangents vs. frequency for a variety of samples with varied sizes of Fe_3O_4 nanoparticles; (b) return loss variation vs. frequency for 4 samples with different nanoparticle sizes in magnetic fluid. MF1, MF2, MF3, MF4 are magnetic fluids with 10 nm, 12 nm, 16 nm, and 17 nm Fe_3O_4 nanoparticles, respectively [122].

4.2 Magnetite (Fe_3O_4) nanoparticles composite for microwave absorption applications

With rapid advancements in science and technology, the use of RF and microwave electronics have increased many folds, which creates electromagnetic interference (EMI) to not only impact human health but also interfere with electronics nearby [128]. Thus, electromagnetic (EM) absorption materials at RF and microwave frequencies have garnered a great deal of attentions because of their application in wireless data communication, radar system and other area networks [126]. For good microwave absorption properties, impedance matching between air and absorbing material as well as reflection loss are very important. Materials that have both desired magnetic and dielectric properties serve this purpose well [129]. Currently, soft magnetic

ferrites and nanomaterials have widely explored for microwave absorption because of their high magnetic, electric and loss properties [130, 131]. Fe_3O_4 is well known for its chemical stability and tailorable magnetic/dielectric losses at microwave regions. Developing low-density composites of high dielectric and magnetic losses as absorbing materials is an effective approach for fulfilling EM absorption performance.

In 2007, Zhou *et al.* reported microwave absorption properties of $\text{SiC@SiO}_2@$ Fe_3O_4 hybrids for the frequency range of 2–18 GHz for samples of different thickness [132]. SiC@SiO_2 nanowires were synthesized using carbothermal reduction method. $\text{SiC@SiO}_2@$ Fe_3O_4 hybrids were produced by adding iron precursor, $\text{Fe}(\text{acac})_3$ to the SiC@SiO_2 suspension in triethylene glycol, which is then heated to 280°C under an argon atmosphere. Microwave absorption properties were investigated for different SiC@SiO_2 to $\text{Fe}(\text{acac})_3$ mass ratios. The EM absorption performance was enhanced by attaching Fe_3O_4 nanoparticles to SiC@SiO_2 nanowires. Specially for 1:3 mass ratio of SiC@SiO_2 to iron (III) acetylacetonate, the measured microwave absorption for a 2-mm thick sample exhibited a minimum reflection loss of -39.58 dB at 12.24 GHz.

Figure 10 shows reflection loss of SiC@SiO_2 and $\text{SiC@SiO}_2@$ Fe_3O_4 hybrids with different mass ratios [132].

In 2007, Qiao *et al.* studied nanochains yolk-shell $\text{Fe}_3\text{O}_4@$ N-doped carbon as a novel microwave absorption material for the frequency range of 2–18 GHz [128]. First, the core-shell $\text{Fe}_3\text{O}_4@$ P(EGDMA-MAA) were synthesized using magnetic field induced precipitation polymerization method, then core-double-shell $\text{Fe}_3\text{O}_4@$ P(EGDMA-MAA)@PPy nanochains were synthesized using oxidant-directed vapor-phase polymerization process. Finally, yolk-shell $\text{Fe}_3\text{O}_4@$ N-doped carbon nanochains were synthesized by carbonized $\text{Fe}_3\text{O}_4@$ P(EGDMA-MAA)@PPy nanochains utilizing salt crystallization method. It was reported that 20% loading of yolk-shell $\text{Fe}_3\text{O}_4@$ N-doped carbon with paraffin-based composites have exhibited strong

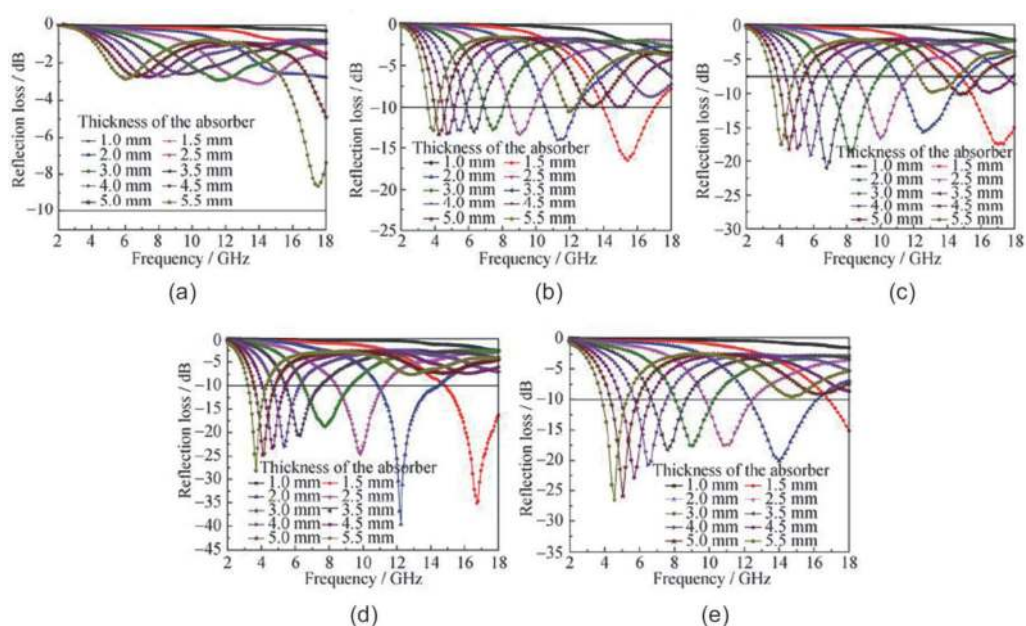
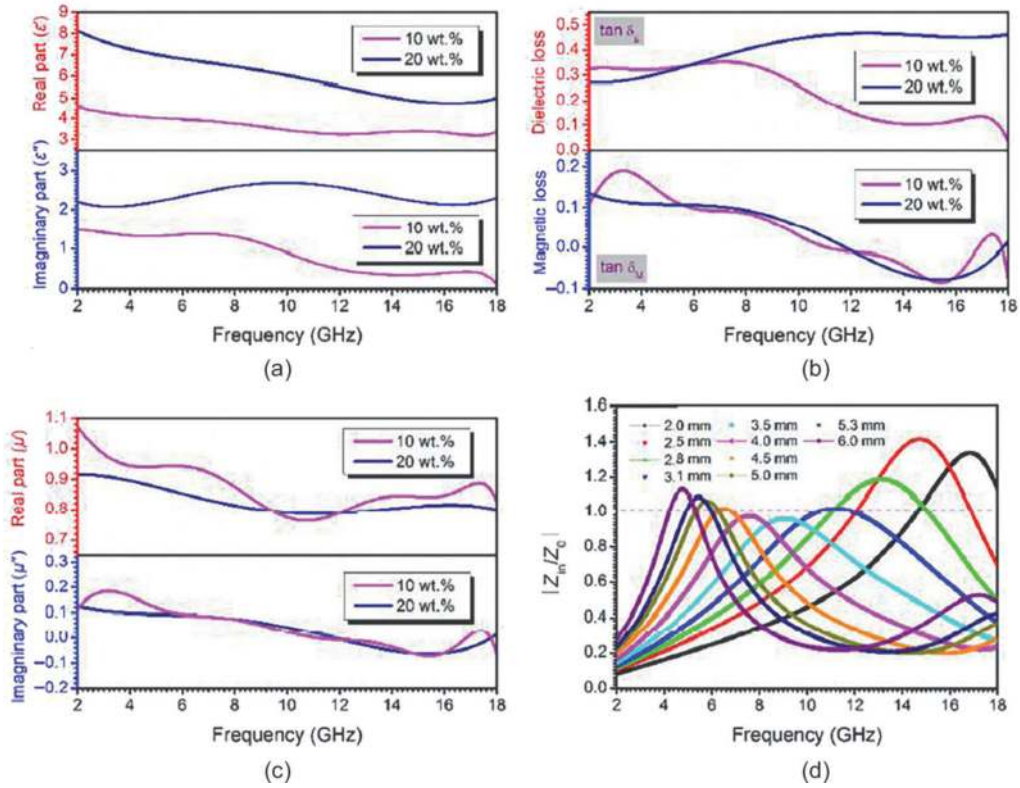


Figure 10. Calculated reflection loss of (a) SiC@SiO_2 nanowires; (b) $\text{SiC@SiO}_2@$ Fe_3O_4 hybrids in ratio 1:1; (c) 1:2; (d) 1:3; (e) 1:4 [132].


Figure 11.

Electromagnetic parameters of $\text{Fe}_3\text{O}_4@N$ -doped carbon nanochains including: (a) complex permittivity; (b) dielectric and magnetic loss; (c) complex permeability; and (d) relative input impedance at different thickness layers [128].

absorption capability with a low reflection loss of -63.09 dB at 11.91 GHz. **Figure 11** shows the complex permeability and permittivity for 10 wt % and 20 wt% loading yolk-shell $\text{Fe}_3\text{O}_4@N$ -doped carbon samples and relative input impedance for samples with different layer thicknesses [128].

The frequency dependent complex relative permeability is given by Eq. (1) [133],

$$\mu_r = \mu' - j\mu'' \quad (1)$$

where μ_r is the ratio of the relative permeability of the material versus that of the free space (μ_0). μ' and μ'' are real and imaginary parts of the relative permeability, respectively.

The magnetic loss tangent is the ratio between the real and imaginary parts given by Eq. (2),

$$\tan \delta_m = \frac{\mu''}{\mu'} \quad (2)$$

The frequency dependent relative complex permittivity can be given by Eq. (3) [133],

$$\varepsilon_r = \varepsilon' - j\varepsilon'' \quad (3)$$

where ϵ_r is known as ratio of the permittivity of the material versus that of the free space (ϵ_0) and ϵ' and ϵ'' are real and imaginary parts of the complex permittivity.

The dielectric loss tangent is given by Eq. (4),

$$\tan \delta_e = \frac{\epsilon''}{\epsilon'} \quad (4)$$

The samples with 20 wt% loading showed the highest relative permittivity (real part) along with high dielectric loss tangent over the entire frequency range, which can be ascribed to the conductive loss inside the nanochain during the propagation of electromagnetic wave through the yolk-shell structure. Due to the geometry of yolk-shell structure, such as high porosity and void spaces, multiple scattering and reflections are generated through the interface polarization, which influences the dielectric loss of the nanochains [128]. It was concluded that high magnetic losses (due to natural resonance and eddy current effect) and dielectric losses (due to interfacial polarization) can be achieved by designing porous magnetic cores with proper yolk shell structure. Hence, better microwave absorption performance can be achieved even at low filler loadings.

Similar prior works using Fe_3O_4 nanoparticles as core material have reported recently. **Table 3** tabulated the microwave absorption performance of Fe_3O_4 nanoparticles-based nanocomposites used with different structures.

4.3 Magnetite (Fe_3O_4) nanoparticles composite for antennas in RF and microwave regions

Tunable electromagnetic properties of nanomaterial-based nanocomposite are key enabler for RF and microwave applications. Several reports have described the development of RF and microwave device applications, such as antennas, and inductors using commercially available dielectric and semiconductor-based substrates. For tunable electronic devices, magnetic nanocomposites can facilitate in designing of fully tunable and magnetically controllable devices. This kind of application requires antennas and other RF devices to be operating at different frequencies or meeting other performance needs such as antenna bandwidth and efficiency. RF devices that are frequency agile or dependent are highly desirable for biomedical and defense applications. Tuning of different parameters of device such as frequency can

Absorbers	Absorber Thickness (mm)	RL _{min} vs. (frequency)	Absorption bandwidth (GHz)	References
PANI/ Fe_3O_4	1.4	-18 dB (8.6 GHz)	—	[134]
ACV/ Fe_3O_4	2	-30.7 dB (16.4 GHz)	8.2	[131]
RGO/SiO ₂ / Fe_3O_4	4.5	-56.4 dB (8.1 GHz)	7.1 (3 mm)	[135]
Fe_3O_4 /RGO	3.5	-45 dB (8.96 GHz)	3.2	[126]
Fe_3O_4 /ZnO	3.5	-22.7 dB (13 GHz)	5.9	[136]
Fe_3O_4 /Ppy/CNT	3	-25.9 dB (10.2 GHz)	4.5	[137]
Fe_3O_4 /C	2.9	-46 dB (12.8 GHz)	6.5	[138]
Fe_3O_4 /TiO ₂	2	-23.3 dB (7 GHz)	5.5	[139]

Table 3. Microwave absorption performance of Fe_3O_4 nanoparticles-based nanocomposites.

be achieved by various methods. One such method for controlling the performance of RF microwave devices is employing tunable magnetic materials such as Fe_3O_4 nanoparticles nanocomposite as the base substrates.

Morales *et al.* in 2011 and 2014 reported implementation of magnetite (Fe_3O_4) nanoparticles and -polydimethylsiloxane (PDMS) magnetodielectric composite for RF and microwave applications [140, 141]. In this work, sub-10 nm Fe_3O_4 nanoparticles have been synthesized using thermal decomposition method. Negligibly low hysteresis losses were reported at room-temperature. Magnetic and dielectric properties of Fe_3O_4 -PDMS nanoparticles composite were extracted using multilayer microstrip line test fixtures with and without an external magnetic field of varied levels. Three different concentrations (30, 50 and 80 wt%) of Fe_3O_4 nanoparticles filler in PDMS matrix were studied for both dielectric and magnetic measurements. A relative permeability of 2.5 along with a magnetic loss tangent of 0.15 at 4 GHz was reported for 80 wt% nanocomposite without the application of external magnetic field [141]. Similarly, a permittivity of 2.8 with a dielectric loss tangent of 0.18 were reported for sample with 80 wt% loading at 4 GHz. **Figure 12** shows the room temperature magnetization (M-H) curve for Fe_3O_4 nanoparticles and Fe_3O_4 -PDMS nanoparticle composites at three different nanoparticles loading (30, 50 and 80 wt%) [141]. No magnetic hysteresis was observed for all three concentrations of Fe_3O_4 -PDMS nanoparticles composite, which is a desirable property for RF microwave antenna applications.

Enhanced permeability and permittivity values of 3.55 and 2.79 along with low magnetic and dielectric loss tangents of 0.02 and 0.019, respectively, were measured for samples with a high loading ratio (80 wt%) of Fe_3O_4 nanoparticles for the composite samples under an external applied field of 0.2Telsa. Based on the optimal magnetic and dielectric properties of nanocomposite under external field polarization, the Fe_3O_4 -PDMS nanocomposites have been used to form the substrate for miniaturized multilayer patch antennas with a center frequency of 4GHz, which showed 58% bandwidth enhancement and 57% of size reduction as compared those of PDMS substrate based counterparts. Meanwhile, a return loss of -23 dB and an antenna gain of 2.12 dBi have been achieved. **Figure 13** shows the schematic of multilayer microstrip patch antenna designed with a Fe_3O_4 -PDMS composite substrate with a 80 wt% Fe_3O_4 filler loading [140].

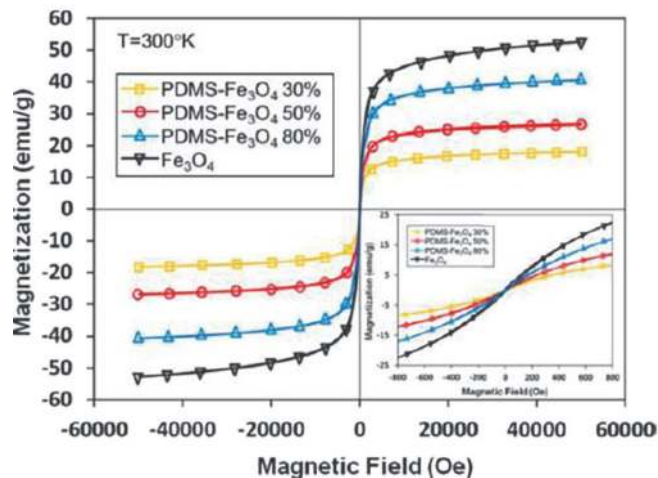


Figure 12. Magnetization vs. magnetic field (M-H) curve for Fe_3O_4 nanoparticles and Fe_3O_4 -PDMS nanoparticles composite at three different nanoparticles loading (30, 50 and 80 wt%) [141].

In 2016, Alqadami *et al.* also reported similar results based on Fe_3O_4 -PDMS nanoparticle composite as magneto-dielectric substrate for MIMO antenna array [142]. The reported antenna was designed with 35%- 65% Fe_3O_4 nanoparticles to PDMS polymer ratio over the frequency range of 5.33–7.70 GHz. The reported results showed a bandwidth enhancement of 40.8% and 57% enhancement in radiation efficiency along with an antenna gain of 9.95 dB gain and a return loss of -33 dB. **Figure 14** shows the fabricated prototype of 2x4 MIMO antenna array [142].

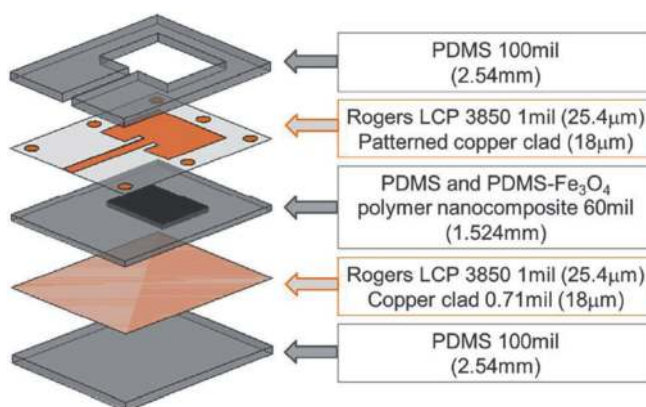


Figure 13. Real permeability of Fe_3O_4 -PDMS nanoparticles composite at varied concentrations of Fe_3O_4 nanoparticles under application of external magnetic field [140].

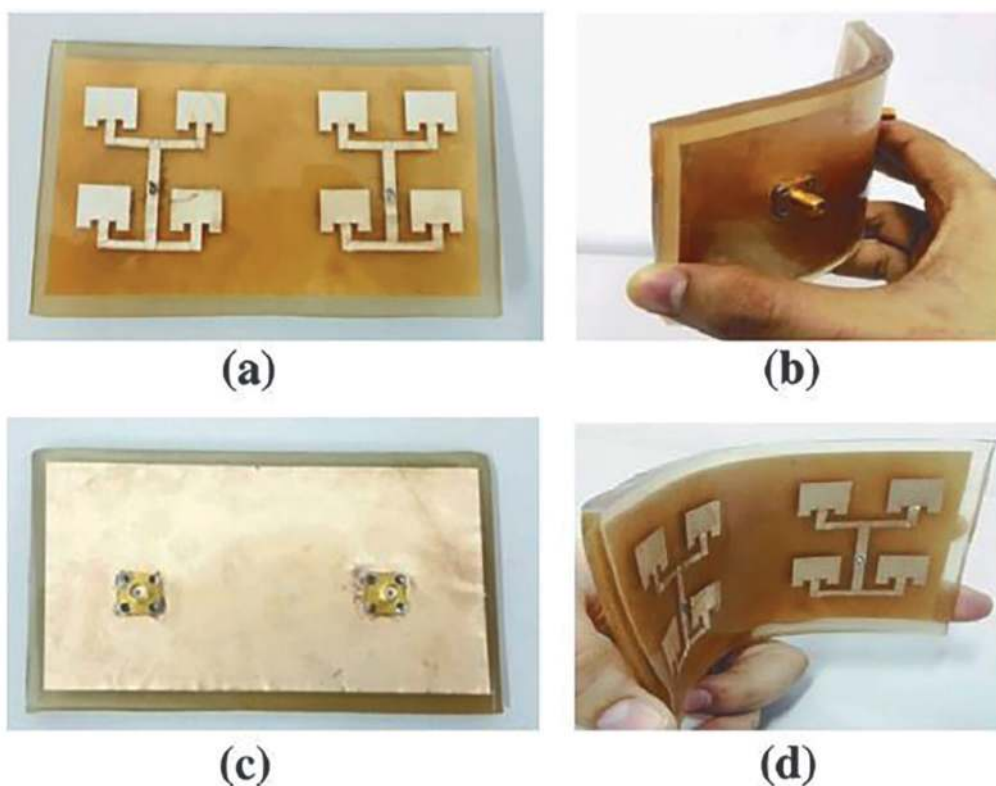


Figure 14. (a) Front view; (b) bending view; (c) rear view, and (d) front bending view for 2x4 MIMO antenna array [142].

Vaseem *et al.* in 2018 developed Fe_3O_4 nanoparticles based magnetic ink and screen-printed nanocomposite substrates for tunable radio frequency devices [31]. The Fe_3O_4 nanoparticles were synthesized using co-precipitation method. Then, prepared Fe_3O_4 nanoparticles were mixed with SU8 polymeric resin, and the oleic acid was used for the functionalization of Fe_3O_4 nanoparticles in the SU8 resin matrix for the compatibility. The Fe_3O_4 nanoparticles composite was printed by a manual screen-printing technique over a FR4 microwave laminate. **Figure 15** shows the fabrication steps for the freestanding magnetic substrate and antenna [31]. To evaluate the electromagnetic properties of the nanocomposite, a tunable antenna was fabricated on the screen-printed magnetic composite substrate. Frequency tuning of the fabricated antenna in response to the application of magnetostatic fields was successfully demonstrated. For center frequency of 8 GHz, 12.5% tuning was achieved under a magnetic field strength of 3.7 kOe.

Recently, Menezes *et al.* in 2020 fabricated magneto-dielectric bio-composite Fe_3O_4 nanoparticles based flexible film for antenna devices [120]. The flexible film was developed using biopolymeric matrices chitosan (Ch), cellulose (BC) and collagen (Col). The thermal, dielectric, and magnetic properties of flexible film and their application as antenna were tested by fabricating a microstrip patch antenna. For Ch, BC and Col based Fe_3O_4 nanoparticles film, dielectric properties were measured in the range of 5.2–8.3, 6.7–8.4 and 5.9–9.1, respectively from 0 to 5 GHz frequency range. The resonance frequency shifted from 4.66 GHz to 5.89 GHz for different weight percentages of Fe_3O_4 nanoparticles in different polymer matrix. For example, the largest tunability in resonance frequency from 5.55 to 4.69 GHz was observed, by increasing Fe_3O_4 nanoparticles amount from 0 to 80% in Ch matrix with return loss lower than 10 dB for all antenna measurements. The enhancement in bandwidth from 3.37 to 6.34% was reported for all antennas. It was demonstrated in the report that the operating frequency of the devices, size and bandwidth can be modulated by varying the substrate composition, and by controlling the magnetic and dielectric losses of the substrate.

Similar works have been reported by Ghaffar *et al.* in 2018 [143], Cannamela *et al.* 2020 [144], Alqadami *et al.* in 2018 [34] Caprile *et al.* in 2012 [35] by fabricating and

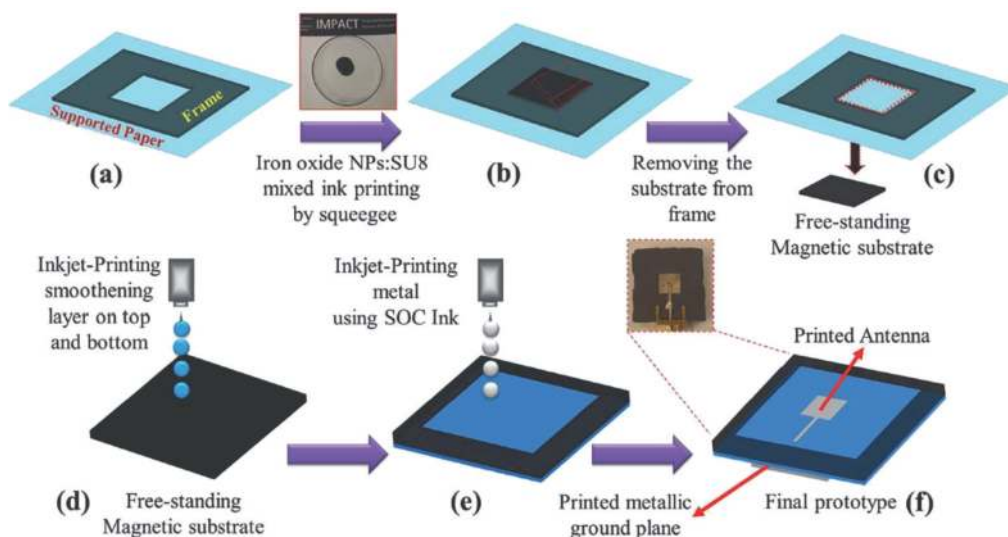


Figure 15. Step-by-step fabrication process flow of magnetic substrate and printed antenna [31].

characterization of frequency tunable patch antennas using printable inks loaded with Fe_3O_4 nanoparticles.

4.4 Magnetite (Fe_3O_4) nanoparticles composite for circulators and inductors in RF and microwave regions

Ferrites and as magnetite nanoparticle composites have also been used extensively in RF and microwave applications like inductive component, isolators, or as circulators [145, 146]. These devices in electronic industry highly depend on the magnetic properties of the material used. The applications based on soft magnetic ferrite materials take advantage of the fact that spin rotation of these materials changes with the direction of external magnetic field. For one direction, ferrites will absorb the microwave field, and for opposite direction it will transmit the field. This non-reciprocal behavior is the basis of devices such as isolators and circulators [20]. Mostly, Ni-Zn and Mn-Zn ferrites are commonly used for such applications, since they are capable

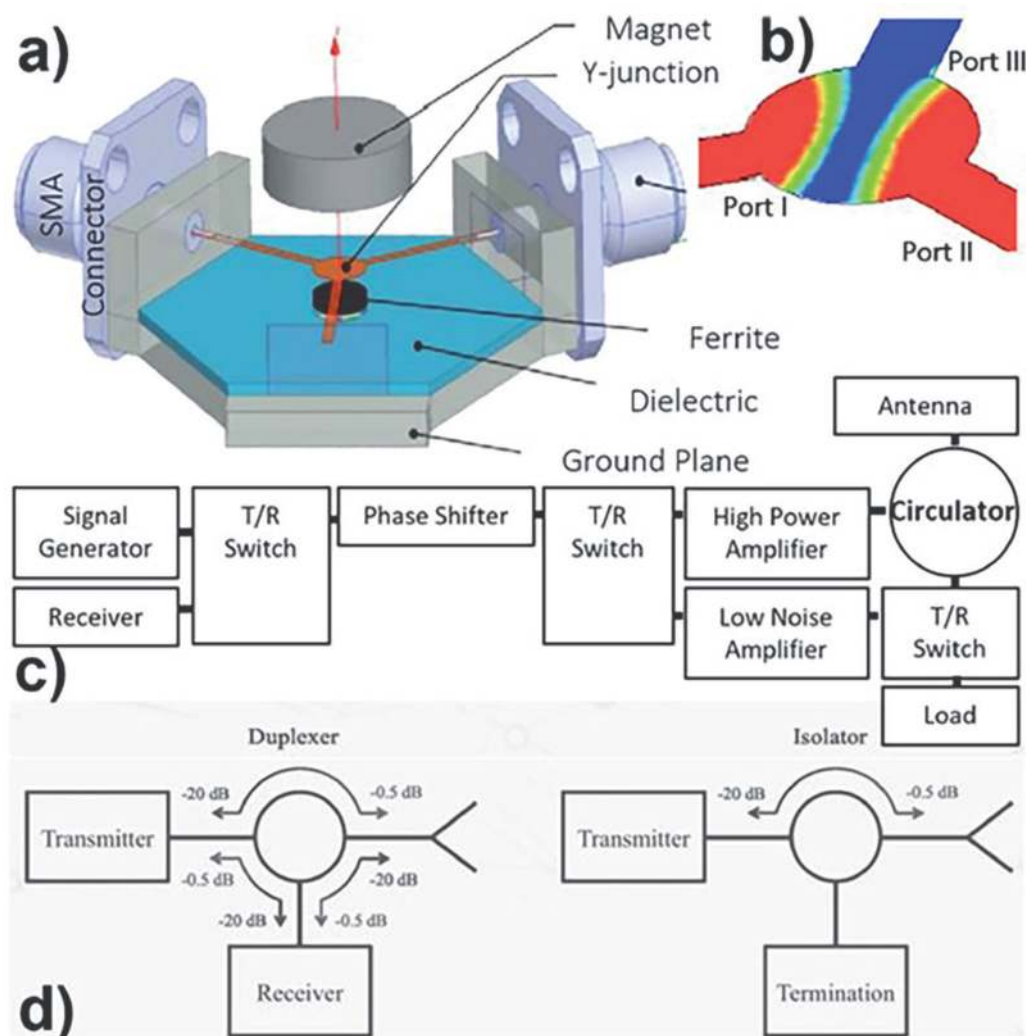


Figure 16. (a) Design of circulator with use of ferrite in it; (b) electromagnetic simulation of circulator; (c) use of circulator in receiver and transmitter module; and (d) circulator as duplexer and isolator [148].

of providing high permeability, low magnetic loss tangent, high stability, and high resistivity. Nevertheless, they typically exhibit high magnetic losses at higher operating frequencies.

Fe₃O₄ nanoparticles based soft magnetic ferrites can be used for non-reciprocal device applications (e.g., isolators and circulators), because Fe₃O₄ nanoparticles with well controlled particle sizes can offer low magnetic and dielectric losses due to their superparamagnetic property at room temperature. In 2017, Sahasrabudhe *et al.* designed the wideband lumped element circulator based on Fe₃O₄ ferrite and reported 125% improvement in terms of bandwidth at 915 MHz center frequency [147]. There is still limited research on the use of Fe₃O₄ nanoparticles for circulator and inductor applications. **Figure 16** shows use of ferrite material for the design and implementation of circulator along with location and functionality of a circulator within a front-end transmit and receiver module, respectively [148].

5. Conclusion

The chapter presents a review of the key synthesis techniques for magnetite (Fe₃O₄) nanoparticles and their applications. Fe₃O₄ nanoparticles have a large area of applications in different fields such as magnetic separation, storage, biomedical applications, catalyst, water purification, electronics, and so on. It was concluded from the synthesis methods that their structural and magnetic properties are highly dependent on the shape and size of the nanoparticles. The morphology of the particles can be controlled by different synthesis parameters. Among the chemical methods, chemical co-precipitation method is the most advantageous due to the ease of the synthesis approach. Improvement in the stability of Fe₃O₄ nanoparticles with appropriate agents is also discussed in the article. With this regard, the current applications of Fe₃O₄ nanoparticles for RF and microwave applications have been discussed. It is important to tune and tailor control suitable particle size with optimized synthesis approach and applied field strength for the design of RF/microwave devices and other applications like hyperthermia and drug delivery. For future application of Fe₃O₄ nanoparticles in biomedical device and electronics applications, it is crucial to not only control the morphology and magnetic properties of the nanoparticle but also optimize synthesis methods to increase the yield on industrial scale. Though there are limited studies presently, applications of Fe₃O₄ nanoparticles in RF/Microwave devices is an emerging area, where new application will be discovered in near future. This will open up new avenues in many sectors including biomedical devices.

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