Spinel Ferrite Nanoparticles:
Synthesis, Characterization and Applications

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ABSTRACT

Spinel ferrite nanoparticle have attracted a great attention due to its widely used in several areas such as in electronics devices, biomedical, catalyst and wastewater treatment. The synthesis, characterisation and application of spinel ferrite nanoparticles in various fields is focuses in this review paper. The most commonly used synthesis methods along with their advantages and limitations were discussed. The most common characterisation techniques and their applications such as power applications, biomedical applications, inductors, high frequency and Electromagnetic Interference (EMI) Suppression are briefly reviewed.

KEYWORDS: Spinel ferrites, Synthesis, Characterization, applications

1. INTRODUCTION

The spinel ferrites are of great interest in basic science, especially for addressing the fundamental relationships between magnetic properties and their crystal chemistry and structure. Crystal chemistry shows how the chemical composition (chemical formula), internal structure and physical properties of minerals are linked together. Spinel ferrites have been investigated in recent years for their useful electrical and magnetic properties and applications in information storage systems, magnetic bulk cores, magnetic fluids, microwave absorbers and medical diagnostics. Ferrite nanoparticles belong to a major group of magnetic nanoparticles and have received a considerable attention due to their wide applications in different fields ranges from biomedical to industrial.

Spinel ferrite nanoparticles are metal oxides with spinel structure of general formula AB_2O_4, where A and B are metallic cations located at two different crystallographic sites, tetrahedral (A site) and octahedral (B site). The cations of both positions are tetrahedrally and octahedrally coordinated to oxygen atoms, respectively (Fig.1). The chemicals to be named as ferrite, at least, there must be Fe (III) in the chemical formula. The common spinel ferrites are MFe_2O_4 (where M = Fe, Co, Mn, Zn, Cu and Ni), and most of them show super paramagnetic properties at the size below or about the 20 nm in diameter. In one unit cell of spinel ferrite, there are 64 tetrahedral and 32 octahedral positions are available for cations, out of which only 8 tetrahedral and 24 octahedral are occupied by cations. The distributions of each metal cations over both sites (A and B sites) are depend on their affinity for both positions. Which depends on the ionic radii of the specification, stabilisation energy, size of the interstices, the synthesis method and the conditions employed during synthesis time. The cations distribution at tetrahedral and octahedral sites have a significant effect on the physical and chemical properties of ferrite nanoparticles. For example, the magnetic properties of ferrite nanoparticles are directly proportional to the distribution and types of the cations at octahedral and tetrahedral sites of the spinel structure. Because, the net magnetic moment of ferrite nanoparticles is the difference between the two sites [1].

The spinel ferrites are unique materials exhibiting ferrimagnetic and semiconductor properties and can be considered as magnetic semiconductors. These materials has been extensively used in several applications including recording heads, antenna rods, loading coils, microwave devices, core material for power transformers in electronics and telecommunication applications [2–4]. Nanosized ferrites may have extraordinary electric and magnetic properties that are comparatively different from

microstructured materials, tailoring them to modern technologies, as well as providing novel applications such as ferrofluids [4], magnetic drug delivery [5], high density information storage [6], photocatalysis [7], gas sensors [8], etc.

Ni–Zn spinel ferrites are magnetic materials that are mostly used in the modern electronics industry. This is due to their high electrical resistivities, which implies low eddy current losses that becomes significant at higher frequencies of electromagnetic fields. Nickel–zinc ferrites also have a very high values of saturation magnetization (Ms) and magnetic permeability, high mechanical strength, low coercivities, low dielectric losses and good chemical stability .The current interest in making nanosized nickel- zinc ferrite particles is to reduce energy losses linked with bulk powders. Also more electronic applications require these materials be pressed into larger shapes with near theoretical density, which is difficult to obtain if the particles have a wide size distribution.[9].
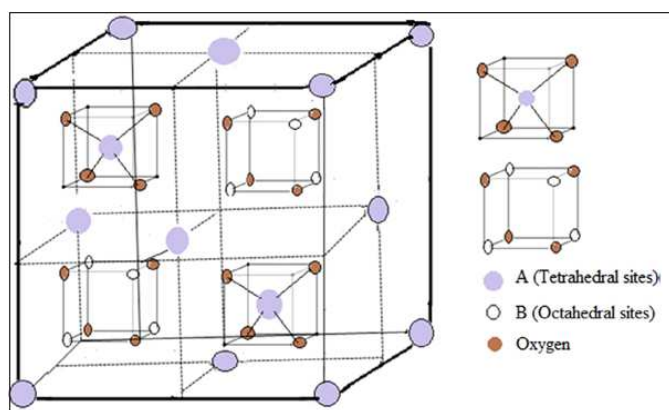

Fig.1. Crystal structure of Spinel ferrite showing tetrahedral and octahedral sites

2. Synthesis methods of ferrite nanoparticles
Recently, a number of synthesis techniques have been developed for the preparation of ferrite nanoparticles, but to date there is no anyone universal method of synthesis of spinel ferrite nanoparticles and each synthesis methods has advantages and also disadvantages. These synthesis methods are grouped into two broad categories namely ''top-down'' and ''bottom-up''. During the ''top-down'' technique materials are pulverised to form tiny particles while in ''bottom-up'' the ions are chemically combined to form the particles. There are several synthesis methods for the ''bottom up'' option includes co-precipitation, solvothermal, hydrothermal, sol-gel, flame spray pyrolysis, thermal decomposition, vapour deposition, sonochemical, microwave-assisted, polyol and microemulsion, techniques. The ''top-down'' synthesis techniques common for, the mechanical milling and pulsed laser ablation. These synthesis techniques have a great advantages such as higher yields, shorter reaction time, improved selectivity, milder reaction conditions and clean reaction [10]. Some of these synthesis techniques are reviewed by Kefeni et al. [1].

2.1. Co-precipitation
Synthesis of spinel ferrite nanoparticles by co-precipitation method is the most convenient, economic, and less time-consuming, has high mass production, and is frequently employed among all other methods in order to achieve uniform-sized particles. In this method, an aqueous solutions, which containing the mixture of divalent and trivalent transition metals, were mixed together in mole ratio of 1:2, respectively. Salts containing Fe(III) are usually used for the source of trivalent metal ions and the experiment is normally conducted in an alkaline medium .By adjusting and controlling the pH of the solution may synthesized super quality ferrite nanoparticles. The pH of the solution is usually adjusted using ammonium solution. The co-precipitation method used in several research studies, for the synthesis of spinel ferrite nanoparticles [11].

2.2. Hydrothermal
In this synthesis method, soluble salts containing divalent and trivalent transition metals are dissolved separately and mixed in the mole ratio of 1:2, respectively [12]. Then add drop-wise organic solvents such as ethylene glycol or ethanol in the mixtures of aqueous solutions and under continuous stirring in order to made homogeneous solution. After that the solution is heated under the high pressure in an autoclave. The hydrothermal method is one of the most promising methods for the large-scale production of ferrite nanoparticles. The synthesis of a good quality of nanferrites with controlled size and size distribution can be obtained by selecting the proper mixture of solvents and varying parameters such as pressure, temperature and reaction time.

2.3. Sol-gel Method
The sol–gel method is widely used for the synthesis of the spinel ferrite nanoparticles, in which metal alkoxide solution undergoes hydrolysis and the condensation polymerization reactions to formed gel and any volatile impurities were removed by the heat treatment after the synthesis reaction. The advantages of this method is, it is low-cost, simple, achievable of narrow particle size distribution, carried out at also at low temperature without the need of any special equipment. However, care is taken for a better control of reaction parameters such as concentration of sol, stirring rate and annealing temperature. The major limitation is the lack of purity in the final product and thus required thermal treatment after the synthesis of ferrite sample to attain high purity. Nevertheless, composition control, particle size, homogeneity and particle distribution can be achieved by sol–gel method. The detailed process for synthesis of Ni-Zn Spinel ferrite can be described as follows (Fig. 2)[13].

2.4. Thermal decomposition Method
The thermal decomposition method is one of the simplest method for the synthesis of spinel ferrite nanoferrites. This method involves thermal decomposition of organometallic precursors such as metallic acetylacetonates and carbonyls in the presence of organic solvents and surfactants for the synthesis of ferrite nanoparticles. On the basis of type of precursor materials, the high or low temperature can be used. The shape and size of ferrite nanoparticles can be controlled by adjusting the temperature [14].
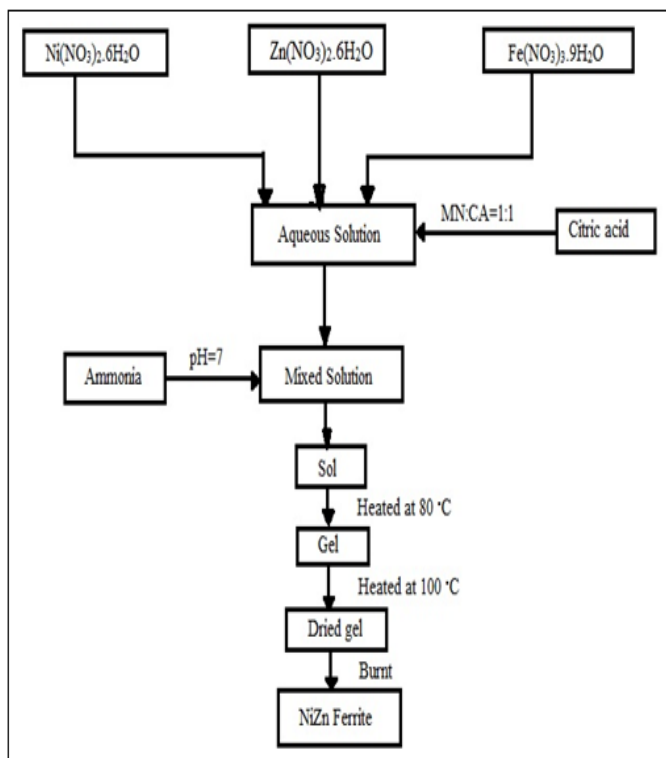
Figure2. Schematic of synthesis of Ni-Zn Spinel ferrite by sol-gel route

2.5. Solvothermal Method
In solvothermal synthesis method, an aqueous or non-aqueous solvents are used to synthesise nanoparticles with precise control over the crystalline phases shape and size distribution. These physical characteristics are altered by changing certain parameters, such as the reaction time, recation temperature, surfactant, solvent and precursors. A number of spinel ferrites and their composites have been synthesized using the solvothermal method. The solvothermal method is useful where the preparation of spinel ferrites with improved chemical and physical properties applicable to both biomedical and industrial areas is required. Due to its simplicity in terms of its reaction, solvothermal method use for the scalable production of spinel ferrites with controlled size and shape [15].

2.6. Sonochemical
The sonochemical route is widely used for the synthesis of spinel ferrite nanoparticles due to facile control of reaction conditions. This method mainly used for controlling the particle size distribution and the achieving homogeneous mixing. Therefore, the temperature and intensity of ultrasonic waves are the major factors which have a direct impact on the particle size of spinel ferrites. During the synthesis process, due to ultrasonic wave, the bubbles are formed in solvent medium and spinel ferrites undergo in situ calcination due to induced high-energy collisions between the ferrite particles. Due to ultrasonication, mixing at the atomic level can be achieve, so that formation of crystalline phase is achievable by annealing at relatively low temperatures [16].

2.7. Microwave assisted Method
The microwave-assisted synthesis method is the recent technique, for the preparation of ferrite nanoparticles. In a microwave system, energy is directly delivered to materials through the molecular interaction with EMR. Conversion of electromagnetic energy into thermal energy generate heat.

Typically, the temperature ranges in microwave-assisted synthesis method from 100^0C to 200^0C and with the shorter reaction time. Vapour produced during the heating time can be eliminated by means of an exhaust drain. The microwave-assisted synthesis method offers an opportunity to synthesize ferrite nanoparticles on a large scale but due to low yield compared to thermal or hydrothermal decomposition methods and coprecipitation method [17].

2.8. Microemulsion Method
In the microemulsion synthesis technique, thermodynamically stable isotropic dispersion of two immiscible liquids stabilised by surfactant molecules is usually used. For example, use of polyoxyethylene ethers as a non-ionic surfactant, n-butanol as a co-surfactant and n-hexane as an oil phase. One of the main advantage of the microemulsion synthesis technique is, it offers the possibility of synthesis diversity of ferrite nanoparticles by changing the nature of surfactant, co-surfactant, reaction conditions and oil water ratio. [18].

2.9. Electrochemical
The electrochemical synthesis method of nanoparticles is similar to coprecipitation method, the difference between these two methods is that the ion sources are due to the scarification of anode electrode through oxidation process. Once the electrode (anode) is oxidised and released into surfactant solution, ions are interacting between electrode-electrolyte interface and formed the corresponding ferrite nanoparticles. The highly pure product and the particle size control are the some advantages of electrochemical method over the other synthesis methods. Purity of the sample and the particle size achieved by optimising the distance between the electrodes or controlling the current [15]. Particularly, in an aqueous solution, current density and the distance between the electrodes considerably influence crystallinity and the particle size. The crystallinity of the synthesized particles could be improved by reducing the distance between the electrodes. Hence, either by increasing the current or by decreasing the distance between the electrodes, particle size tends to enhance. However, factors such as pH, current density, electrolyte concentration,, distance between the electrodes should be properly and electrode choice calculated in order to obtained the required quality of ferrite nanoparticles intended to be synthesised [19-20].

2.10. Mechanical milling Method
Mechanical milling is a typical method for the preparation of nanoferrites by using a high-energy shaker, tumbler mills or planetary balls. This method is an example of the "top-down" approach for the synthesis of spinel ferrite nanoparticles. The major advantage of mechanical milling method is that it offers the possibility of scaling up to tonnage quantities of the materials for various applications while its major drawback is that the contamination of nanoparticles during milling [21]. This synthesis method is significantly used in order to obtain nanoferrites with a wide size distribution of 35nm –85 nm.

3. Characterization
There are so many techniques are used to characterized the nanoferrites. These techniques are varying in sensitivity and they provide quality of the information about the sample. The characterization techniques which are most common

includes: X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), superconducting quantum interference device (SQUID, vibrating sample magnetometer (VSM),), UV- Visible, energy dispersive X-ray spectroscopy (EDS), Fourier transform infrared (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS), Mössbauer spectroscopy (MS), thermo gravimetric analysis (TGA), dielectric magnetic force microscopy (MFM) and electron paramagnetic resonance. In this review paper the major characterisation techniques were briefly discussed based on their application areas.

3.1. XRD

X-ray diffraction (XRD) is a powerful nondestructive technique for characterizing crystalline materials. It provides information on structures, phases, preferred crystal orientations (texture), and other structural parameters, such as average grain size, crystallinity, strain, and crystal defects. X-ray diffraction peaks are produced by constructive interference of a monochromatic beam of X-rays scattered at specific angles from each set of lattice planes in a sample. The peak intensities are determined by the distribution of atoms within the lattice. XRD used $CuK\alpha$ as radiation source for extracting XRD patterns of samples. The site radii, hopping bond length, shared and unshared edges are the structural parameters which were also calculated using the X-ray diffraction data.

The crystallite size is estimated by using the Debey Scherrer's formula [24].

$$D = \frac{0.9\,\lambda}{\beta cos\theta} \qquad (1)$$

Where D is the particle size, β is the full-width at half-maximum, λ is the wavelength of x-ray (1.5406 Å); and θ the angle of diffraction.

Induced strain in the NiZn ferrite for different concentration of Gd was calculated from Williamson –Hall method [9].

$$\beta cos\theta = 4\varepsilon sin\theta + \lambda/D \qquad (2)$$

Where D is particle size, λ is wavelength of X-ray, β is FWHM measure in radian, ε is the induced lattice strain and θ is the Bragg Angle. The induced lattice strain obtained using above formula

The X-ray density (ρ_x), bulk density (ρ_m) and porosity (P) of the samples were calculated using the following equations [4].

$$\rho_x = \frac{nM}{N_A\,V} \qquad (3)$$

$$\rho_m = \frac{m}{\pi r^2 h} \qquad (4)$$

$$P = 1 - \frac{\rho_x}{\rho_m} \qquad (5)$$

Where (M) is the molar mass, (m) is the mass of pellets, (r) is the radius of the pellets, N_A the Avogadro's number, Z is the number of molecules per unit cell (Z = 8 for Spinel ferrites), and (V) is the volume of unit cell.

3.2. Morphological Study

The surface morphology of spinel ferrite nanoparticles could be investigated by scanning electron microscopy (SEM) and Transverse electron microscopy (TEM). These techniques

are used in large scale to measure their diameter and to visualise the morphology of ferrite nanoparticles. Particularly, TEM is a more informative technique in providing morphological, compositional, and crystallite of ferrite nanoparticles. TEM provide the better resolution down to atomic scale. Scanning electron microscopy is a surface sensitive imaging technique. It utilizes the electrons generated due to interaction of beam with the sample surface. It is commonly used to study the aggregation state and the surface morphology of nanoparticles. SEM is used frequently than TEM but resolution of SEM is poorer than the TEM shown in fig.3(a) and 3(b)[25].
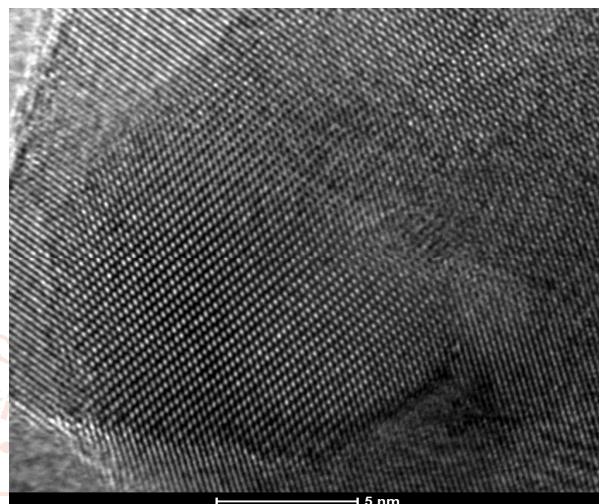

Figure 3.(a) TEM image of $Ni_{0.65}Zn_{0.35}Gd_{0.25}Fe_{1.75}O_4$

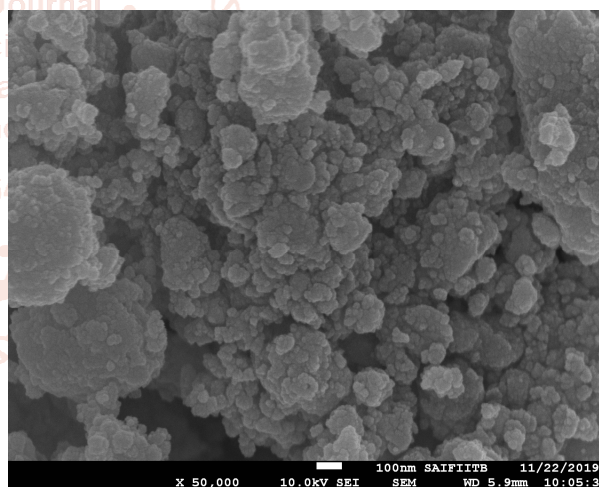Figure 3.(b)SEM image of $Ni_{0.65}Zn_{0.35}Gd_{0.25}Fe_{1.75}O_4$

3.3. Surface area

BET is the characterization technique used for the surface area determination of ferrite nanoparticles. This method involves drying of powder ferrite nanoparticles in a vacuum and then measure the adsorption of nitrogen gas on the surface and micropores. The fundamental assumption in BET characterization technique is that nitrogen gas access to the whole surface of the particles [26].

3.4. Magnetic properties

The common techniques were used to investigate and measure the magnetic properties of ferrite nanopartcles are VSM, EPR and SQUID. EPR is a sensitive method in identification and detection of free radicals and the paramagnetic centres such as F-centre in the chemicals and chemical reactions. This is a alternative method to find the

physical properties of ferrite nanoparticles and their behaviour under the influence of external magnetic field. EPR is based on the interaction of an external magnetic field with the magnetic moments of an unpaired electrons in ferrite sample. Vibrating sample magnetometer(VSM) and Superconducting quantum interference device(SQUID) used where high sensitive measurements of magnetisation are required, sensitivity limit of VSM is about 10^{-6} emu [27]. VSM study investigate the Saturation magnetization (Ms), Coercivity(Hc), Remanence (Mr), squareness ratio(SQR). The variation of saturation magnetization with applied external magnetic field illustrated in Fig. 4. SQUID measures different sample types such as crystals, powders, thin films, gases and liquids. The samples under investigation packed into a gelatin capsules. By using SQUID technique it is possible to determine Ms, Hs and residual magnetisation of ferrite nanoparticles at applied external magnetic field and temperature.
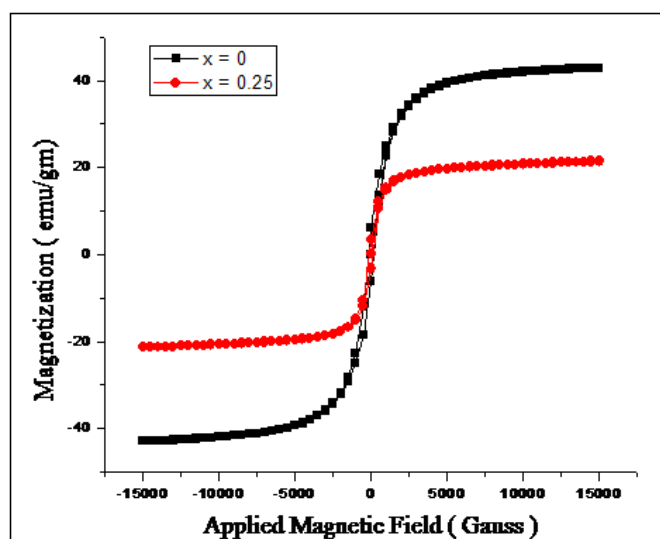

Figure4. Variation in magnetization of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ and $Ni_{0.65}Zn_{0.35}Gd_{0.25}Fe_2O_4$ Ferrites

3.5. Dielectric properties
Dielectric behaviour is one of the most important properties of ferrites which markedly depend on the preparation conditions, e.g. sintering time, and temperature, type and quantity of additives. The study of dielectric properties produces valuable information on the behaviour of localized electric charge carriers leading to greater understanding of the mechanism of dielectric polarisation in these studied ferrite samples. The dielectric properties were studied as a function of temperature, frequency and composition for a spinel ferrites. An "abnormal" behaviour of the dielectric constant is found and also the loss factor as a function of frequency and temperature in comparison with the "normal" behaviour of spinel ferrites [28].

4. Applications of ferrite nanoparticles
Transition metal oxides, particularly, spinel ferrite nanoparticles has been attracted the researchers due to their useful electrical, magnetic, chemical and optical properties. Their application ranges from biomedical to the modern information technologies which include but not limited to satellite communication, antenna cores, memory devices, high-density storage, computer components, microwave absorbing materials, sensors and biosensors, transformer core, magnetic recording media [29].

4.1. Inductors.
Inductors are used as elements in frequency selective circuits. spinel ferrites are primarily used as inductive components in a large variety of electronic circuits such as filters ,low noise amplifiers, impedance matching networks, voltage-controlled oscillators. The basic components to produce the inductance are a very soft ferrite and a metallic coil. To provide high permeability at the operating frequency the ferrite film synthesized by the process compatible with the integrated circuit manufacturing process [29].

4.2. High Frequency
Ferrites have low losses and relatively high permeabilities varies with polarizing fields The magnetic materials having a more demands for high-frequency applications such as telecommunication systems and in radars, as microwave technology requires higher frequency range and bandwidths up to 100 GHz. Ferrites are nonconducting oxides and therefore allow the total penetration of electromagnetic(EM) fields, in contrast with metals, where the skin effect strictly limits the penetration of high-frequency field [30]. At these frequencies, the domain walls are unable to follow the fields and the absorption of microwave power take place by the spin dynamics.

4.3. Power Application
Spinel ferrites were used in Power applications. Power supplies is an integral part of a large variety of devices such as computers, all kinds of peripherals, TV, video systems, all types of small and medium instruments. A compact and efficient power unit is obtained called as switched-mode power supplies (SMPSs). For this application, the mains power signal is first rectified as regular pulses (typically rectangular) at a high frequency and feed into a ferrite transformer, and finally it is rectified again to provide the required power to the given instrument. An increase in power delivery and efficiency can be obtained by increasing the working frequency of the transformer. [29].

4.4. Electromagnetic Interference (EMI) Suppression.
The considerable increase in the amount of electronic equipment such as high-speed digital interfaces in computers and notebooks, scanners, digital cameras and so forth, in small areas, has seriously enhanced the possibility of disturbing each other by electromagnetic interference (EMI). In particular, the fast development in wireless communications has led to interference induced by electric and magnetic fields. Electromagnetic interference can be defined as the degradation in performance of an electronic system caused due to an electromagnetic disturbance [31]. The noise from electric devices is usually produced at frequencies higher than circuit signals. To avoid, or to minimize EMI, suppressors should work as a low-pass filters, that is, circuits block signals with higher frequency value.

4.5. Biomedical applications
The spinel ferrites finds application in the field of nanomedicine is targeted drug delivery using magnetic metal oxide nanoparticles by applying an external magnetic field at the target tissue. The magnetic property of these structures allows controlling the orientation and location of particles by changing the direction of the applied external magnetic field. Pharmaceutical design and research in the field of nanotechnology offer novel solutions for diagnosis and therapies. The magnetic nanoparticles and magnetic

spinel ferrites properties, approaches in magnetic liposomes, magnetic polymeric nanoparticles, MRI, hyperthermia and especially magnetic drug delivery systems, which have recently developed in the field of magnetic nanoparticles and their medicinal applications. Spinel ferrite (SF) have excellent magnetic characteristics, high specific surface area, surface active sites, high chemical stability, tuneable shape and size, and options for functionalization[32].

5. Conclusions

In this paper, synthesis methods, crystal structure, properties, and applications of spinel ferrites are summarized. Spinel ferrite nanoparticles gained a most interesting applications in electronic circuits as inductors, in high-frequency systems, in power delivering devices, in electromagnetic interference suppression, and in biotechnology etc. The specific focus was on the methods of preparation (microemulsion, ultrasonic, coprecipitation, sol–gel, microwave-assisted, non-hydrolytic, solvothermal, mechanical milling, etc.), the crystal structure, magnetic and dielectric and different application of spinel ferrite nanoparticles. The influence of the synthesis method on the particle size of spinel ferrites was also described. The crystal structure, cationic distribution and exchange interaction between the ions are responsible for the magnetic behavior in spinel ferrites. Finally, it can be concluded that spinel ferrites are suitable materials for many technological applications.

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