Green Synthesis of Spinel Ferrite Nanoparticles: A review

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Abstract:

Considering numerous applications in biomedical, electronic devices, wastewater treatment, industries etc. spinel ferrite nanoparticles have becomematerials great interest. Because of its many advantages over conventional synthesis techniques, the green or bio production of ferrite nanoparticles has attracted enough attention recently. The green production of spinel ferrite nanoparticles, their characterisation, and their utilisation in many fields are the main topics of this review article. The use of plant extracts or plant-based synthesis techniques of spinel ferrites is discussed.

Keywords: Spinel Ferrite; Magnetic Nanoparticles; Mix Ferrite; Green Synthesis.

1. Introduction:

Iron oxide (Fe_2O_3) coupled with one or more additional metallic element such as cobalt (Co), nickel (Ni), or manganese (Mn), results in the class of magnetic materials known as ferrites. In many different applications, including magnetic recording media, transformers, inductors, microwave devices, and magnetic sensors, these materials are frequently employed because they have distinctive magnetic properties. Based on their crystal structure, ferrites can be roughly divided into two groups: hexagonal ferrites and spinel ferrites.

(a) Spinel Ferrites:

Spinel ferrites are represented by the general chemical formula MFe_2O_4 and have a cubic crystal structure. M is a divalent metallic cation such as Ni, Zn, Fe, Co, Mn, or Cu. The face-centered cubic (fcc) lattices that make up the structure of spinel ferrites are interpenetrating, and the metal ions occupy both the tetrahedral and octahedral sites in a predetermined sequence. The tetrahedral positions are occupied by the Fe ions, and the octahedral ones by the M ions. Spinel ferrites have the general formula $(M_xFe_{3-x})O_4$, where x can be between 0 and 1.

(b) Hexagonal Ferrites:

The chemical formula for hexagonal ferrites is $MFe_{12}O_{19}$, where M is a divalent or trivalent metal cation like Ba, Sr, Pb, Y, Bi, Co, or Ni. Hexagonal ferrites have a hexagonal crystal structure. The hexagonal close-packed (hcp) array of oxygen ions makes up the structure of hexagonal ferrites, while the metallic ions occupy both tetra and octahedral sites in a predetermined arrangement. The tetrahedral positions are occupied by the M ions, whereas the octahedral sites are occupied by the Fe ions. The typical formula for hexagonal ferrites is stated as MFe_2O_4, where M can be any of the following divalent or trivalent metal ions: Ba, Sr, Pb, Y, La, or other. In general, magnetic features including strong magnetic anisotropy, high coercivity, and high magnetic permeability are present in both spinel and hexagonal ferrites. The magnetic properties of ferrites can be well tuned by controlling various synthesis conditions, such as composition, grain size, and morphology. The broad classification of ferrites is shown in figure 1.
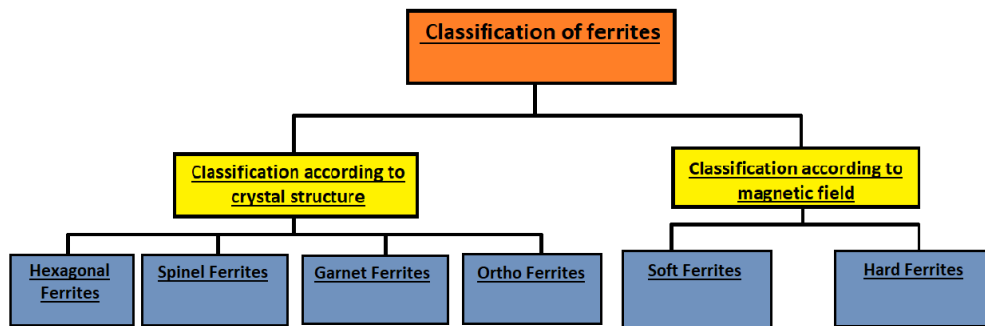

Figure 1: Broad classification of ferrites.

Due to its many advantages over conventional synthesis techniques, the green or bio production of ferrite nanoparticles has attracted enough attention recently. Some of the main benefits of creating ferrite nanoparticles by green or biological means include: friendly to the

environment Utilising organic, non-toxic, and renewable resources like plant extracts, microorganisms, and biowaste as reducers and stabilisers is known as "green" or "bio" synthesis. As a result, there is no longer a need for dangerous chemicals, and the synthesis process has less of an influence on the environment. Cost-effective: Given that it makes use of low-cost starting materials like plant extracts and biowaste, green or biosynthesis of ferrite nanoparticles is a cost-effective process. Additionally, it cuts down on the amount of energy and processing time needed for the synthesis. Control of size and shape: the shape, size, and morphology of the ferrites can be precisely controlled using green or biological synthesis techniques. This is significant because the nanoparticles' shape and size determine their physical and magnetic characteristics.

Green or naturally occurring ferrite nanoparticles are biocompatible and can be employed in a lot of biological applications, including magnetic hyperthermia, medication, magnetic resonance imaging (MRI) etc.Due to the distinct chemical and structural characteristics of the natural reducing agents and stabilisers, green or bio synthesis processes can enhance the magnetic and catalytic properties of the ferrite nanoparticles. In general, the green or bio synthesis of ferrite nanoparticles offers a sustainable and environmentally friendly approach to the synthesis of nanomaterials with many benefits over conventional synthesis techniques. It provides apromising avenue for the development of advanced materials with enhanced properties and biomedical applications.

2. Properties of spinel ferrites:

Due to their special characteristics and prospective uses in numerous industries, including biomedical engineering, catalysis, and magnetic data storage, ferrite nanoparticles have attracted enough attention. Numerous studies have been done recently on the synthesis and characterisation of various ferrite nanoparticle forms.

Ferrite nanoparticles have exceptional magnetic characteristics, which make them appropriate for a lot of uses, including medical applications, magnetic separation, data storage etc. $CoFe_2O_4$ and Fe_3O_4 nanoparticles have received the most attention among the numerous ferrite nanoparticles because of their high magnetization values. Additionally, ferrite nanoparticles have unique optical characteristics that have been researched for use in photocatalysis and sensing. When compared to other ferrite nanoparticles, $ZnFe_2O_4$ and $CuFe_2O_4$ nanoparticles have shown

promising optical properties. Sonia*et al.* (2020) created $ZnFe_2O_4$nanoparticles by the coprecipitation process and attained a maximum absorption peak at 351–398 nm. Contrarily, Dave*et al.*, (2022) reported that the highest absorption peak for $CuFe_2O_4$ nanoparticles they created via the sol-gel approach was ~200-400 nm.

Strong catalytic properties in ferrite nanoparticles have been exploited for a number of processes, including the treatment of wastewater and the synthesis of organic molecules. When compared to other ferrite nanoparticles, $MnFe_2O_4$ and $NiFe_2O_4$ have demonstrated promising catalytic properties. Mandal*et al.*, (2020) created $MnFe_2O_4$nanocomposites and they showed a maximum catalytic efficiency of 84 to 97% for the degradation of the methylene blue (MB) dye.

In addition to the qualities mentioned above, ferrite nanoparticles' thermal stability, biocompatibility, and electrical conductivity have all been investigated. In conclusion, ferrite nanoparticles have shown tremendous potential in a variety of fields due to their particular properties. Numerous studies have been done on the magnetic, optical, and catalytic properties of materials, and $CoFe_2O_4$, Fe_3O_4, $CuFe_2O_4$, $NiFe_2O_4$etc. nanoparticles have shown promise in these fields. In addition, thermal stability, biocompatibility, and electrical conductivity have also been explored, and the reported values suggest that these nanoparticles have excellent potential in many applications.An image showing various properties of spinel ferrites is shown in fig. 2.
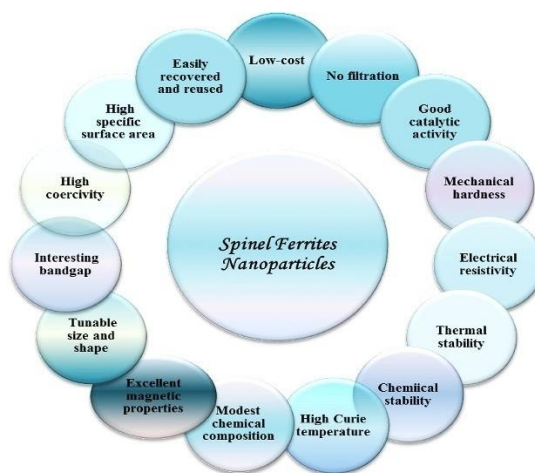

Figure 2: Different properties of spinel ferrites nanoparticles. (**Source: Soufi et al., (2021)**)

3. Chemical synthesis routes of spinel ferrites:

(a) Auto-combustion route:

Preparation of Sol: A sol is prepared by dissolving metal salts, such as iron nitrate and metal-organic compounds, such as citric acid, in a solvent, such as water or alcohol.Fuel Addition: The sol is given a fuel addition, such as urea or glycine. The fuel provides both the necessary heat for the combustion reaction as well as acting as a reducing agent. Heating and Stirring: To evaporate the solvent and create a thick gel, the mixture of fuel and solvent is heated and agitated.Auto-combustion: The metal salts or metal-organic compounds and the fuel undergo an auto-combustion reaction when the viscous gel is heated to a high temperature. This reaction generates a lot of heat, which aids in maintaining the reaction's requisite high temperature. Product Formation: Depending on the initial materials employed, the auto-combustion reaction produces ferrite nanoparticles such as iron oxide (Fe_3O_4) or zinc ferrite ($ZnFe_2O_4$) depending on the starting materials used.Post-Combustion Treatment: The resulting product is typically annealed or calcined to remove any remaining fuel and to enhance its crystallinity and other properties.

(b) Coprecipitation

Solution preparation: Deionized water is used to prepare solutions of metal salts like zinc and iron nitrate. Solution Combination: To create a homogenous solution, the solutions are combined while stirring. Precipitant Addition: The solution is slowly stirred while a precipitating agent, such as ammonium hydroxide, is added to cause the metal ions to precipitate out as hydroxides. Ageing: To allow for more precipitation and particle growth, the final mixture is aged for a predetermined period of time, usually several hours. Filtration and Washing: To remove any contaminants, the precipitate is first filtered to separate it from theaquous solution and then repeatedly washed with the deionized water. Drying: To remove any leftover water, the resulting solid is dried in an oven or under vacuum. Calcination: The dried solid is then calcined at high temperatures, typically 600-1000°C, to form the spinel ferrite phase.

(c) Microwave assisted synthesis

Microwave-assisted synthesis, a relatively new and evolving method for producing ferrites, has many advantages over traditional synthesis methods, including quicker reaction rates, shorter reaction times, and better control over particle size and morphology. Ferrites are produced

using microwave radiation in this method, hastening the heating process and ensuring that heat is evenly dispersed throughout the reaction mixture. The general procedure for synthesising ferrites with microwave aid consists of the following steps: Making the precursor solution The metal salts are combined with other chemicals like chelating agents and surfactants to generate the precursor solution, which is then dissolved in a suitable solvent like water, ethanol etc. Then, the precursor solution is exposed to microwave radiation with a particular power and exposure period. The reaction mixture is quickly heated by the microwave radiation, which promotes the development and nucleation of the ferrite particles. Cooling: The mixture is allowed to cool at room temperature once the appropriate reaction time has been attained. This aids in halting the ferrite particles' future development or aggregation. The synthesised ferrite particles are then filtered out of the reaction mixture and repeatedly washed in water or alcohol to eliminate any impurities or unreacted chemicals. The ferrite particles are then heated to a high temperature and dried at a specified temperature to eliminate any remaining organic compounds and to improve the crystallinity and magnetic properties of the ferrite particles.

(d) Solid state method

For the manufacture of ferrite samples, the solid-state technique is frequently employed. In order to create the appropriate ferrite phase, this approach uses a high-temperature reaction of metal oxide particles. The following steps make up the general process for the solid-state method of synthesis of ferrites: Weighing and mixing: To create a homogeneous combination, metal oxide particles are combined after being weighed and mixed in a ball mill or mortar and pestle. Calcination: To help the combination produce the necessary ferrite phase, the mixture is next heated to a high temperature, usually between 800°C and 1200°C, in a furnace for a predetermined amount of time. Grinding: Using a ball mill or mortar and pestle, the calcined product is subsequently reduced to a fine powder. Pressing and Sintering: The ground powder is then pressed into the desired shape using a machine press or other suitable equipment. The pressed sample is then sintered at high temperatures, typically between 1100°C to 1300°C, for several hours to obtain the final product, depending upon ferrite type.

Table 1: Various synthesis routes of spinel ferrite nanomaterials

S.	Ferrites	Synthesis	Materials	References

N.		Method		
1.	Cobalt Ferrites	Combustion	$Co(NO_3)_2 \cdot 6H_2O$, Glycine $Fe(NO_3)_3 \cdot 9H_2O$,	Houshiar*et al.*, (2014)
2.	Spinel Cobalt Ferrites	Thermal Treatment	Cobalt Nitrate, Iron Nitrate, PVP	Naseri*et al.*,(2010)
3.	Nickel Ferrite	Sol-Gel Method	Nickel Ferrite, Ferric Nitrate, PPA	Chen*et al.*,(2001)
4.	Zn-Doped Nickel Ferrite	Microwave Synthesis	Nickel Nitrate Hexahydrate, Ferric Nitrate Nanohydrate, Zinc Nitrate Hexahydrate, Urea	Sertkol*et al.*, (2009)
5.	Ni-Zn Ferrite	Chemical Coprecipitation Method	$NiCl_26H_2O$, $Zncl_2$, $Fecl_3$	Shahane*et al.*,(2010)
6.	Zn Ferrites	Solid State Combustion Method	$Fe(NO_3)_3,9H_2O$,Zinc Nitrate	Bardhan*et al.*,(2010)
7.	Mn-Zn Ferrites	HydrothermalPrecipitationApproach	$Fe_2(SO_4)_3$, $ZnSO_4$, and $MnSO_4$.	Xuan*et al.*, (2007)
8.	Ni-Zn Ferrites	Co-Precipitation Method	$NiCl_2 \cdot 6H_2O$, $FeCl_3$and $ZnCl_2$	Rao*et al.*,(2006)
9.	Lanthanum DopedMg-Zn Ferrites	Sol-Gel Citrate Nitrate	Zinc Nitrate; Ferric Nitrate, Magnesium Nitrate; Lanthanum Nitrate and Citric	Mullai*et al.*,(2012)
10.	(Mg-Cu)-ZnFerrites	FlameSpray Method	$ZnFe_2O_4$, $Mg_{0.2}Cu_{0.2}Zn_{0.62}Fe_{1.98}O_{3.99;}$ $Mg_{0.5}Zn_{0.5}Fe_2O_4$	A.vital*et al.*,(2007)
11.	Zn Ferrite	Thermal Treatment	Iron Nitrate; Zinc Nitrate,	Naseri*et al.*,(2011)
12.	Co-Zn Ferrites	Sol-Gel Method	Cobalt Nitrate; Zinc Nitrate ;Gadolinium Nitrate; Iron Nitrate	Pawar*et al.*, (2018)
13.	Dy Doped Co-Zn Ferrite	Sol-Gel Auto Combustion	Zinc Nitrate, Cobalt Nitrate, Iron Nitrate, Dysprosium Nitrate Glycine	Kulal*et al.*, (2012)
14.	Cr Doped Lithium Ferrites	Sol-Gel Method	Lithium Acetate, Ferric Nitrate, Chromium Nitrate, 70% ;Glycolic Acid; Ammonia.	Srivastava *et al.*,(2014)
15.	Lithium Ferrite	Auto Combustion Method	Metal Nitrates,Glycine,Citric Acid	Seema*etal.*,(2005)
16.	Copper Doped Lithium Ferrites	Sol-Gel Method	Copper Nitrate Hexahydrate, Lithium Nitrate Hexahydrate, Ferric Nitrate Nanohydrateand Ethylene Glycol	P.Hajasharif *et al.*,(2019)
17.	Tio2 Doped	Microwave	Cobalt III	Magdalane*et*

	Cobalt Ferrites	Method	NitrateHexahydrate,[Fe(NO$_3$)$_3$•9 H$_2$O]	*al.*,(2021)
18.	Co Doped Cu Ferrites	Microwave Assisted Sol-Gel Method	Cu(NO$_3$)$_2$·6H$_2$O; Co(NO$_3$)$_2$·6H$_2$O; Fe(NO$_3$)$_2$·9H$_2$O	Azam*et al.*, (2012)
19.	La Doped Barium Ferrites	Facile Hydrothermal Method	Barium Nitrate,Ferric Nitrate,Lanthanum Nitrate,Sodium Hydroxide	Keerthana*et al.*,(2022)
20.	Gadolinium Doped Cobalt Ferrites	Hydrothermal Process	Fe(NO$_3$)$_3$·9H$_2$O; Co(NO$_3$)$_2$·6H$_2$O	Peng*et al.*, (2011)
21.	HexaFerrites	Hydrothermal Synthesis	Calcium Nitrate,Aluminium Nitrate,UreaFerric Nitrate,	Jinendra*et al.*, (2019)
22.	Nickel Ferrite	Simple Solvothermal Method	Nickel Chloride Hexahydrate; Iron Chloride Hexahydrate	Wang*et al.*,(2009)
23.	Mg Ferrites	Combustion Method	Magnesium Nitrate Hexahydrate; Iron Nitrate Nonahydrate; Urea (CH$_4$N$_2$O),	Nguyen*et al.*,(2019)
24.	Ni-Zn Ferrites	ThermalTreatment Method	Iron (III) Nitrate, Nickel(II) Nitrate, and Zinc Nitrate,	Leng*et al.*, (2019)
25	Cobalt Ferrite	Chemical Precipitation	Fe(NO$_3$)$_3$.6H$_2$O,Co(NO$_3$).4H$_2$O, NaOH,H$_2$O$_2$,Cobalt Nitrate	Uzunoglu*et al.*,(2019)
26	Cobalt Ferrite	Combustion	Analytical Grade Cobalt Nitrate,FerricNitrate,Glycine	Salunkhe*et al.*,(2012)
27.	Mg-Ferrite	Thermal Treatment	Iron Nitrate,Mg Nitrate, PVP	Naseri*et al.*,(2011)
28.	NiSubstituted Cobalt Ferrite	Sol-Gel Auto Combustion	C$_6$H$_8$O$_7$H$_2$O, Co(NO$_3$)$_2$.6H$_2$O,Ni(NO$_3$)$_2$.6H$_2$O; and(Fe(NO$_3$)$_3$9H$_2$O)	Mohd-Hasim*et al.*, (2013)
29.	Co-Zn Ferrites	Microwave Assisted Hydrothermal Method	Cobalt Nitrate,Urea/Ammonia;Nickel Nitrate,Iron Nitrate,	Nabavi*et al.*, (2016)

4. Green synthesis routes of spinel ferrites

Green synthesis approaches of ferrite samples incorporate the use of environmentally safe and sustainable techniques without the use of poisonous and dangerous substances to create ferrite nanoparticles. To create highly stable and biocompatible ferrite nanoparticles, these techniques utilise natural and renewable resources like plant extracts, microorganisms, and other bio-based materials as reducing and capping agents. The following steps make up the general process for

ferrite sample green synthesis routes: choosing a plant extract to create extremely stable and biocompatible ferrite nanoparticles, the plant extract is chosen based on its reducing and capping capabilities. Precursor solution preparation: The metal salts are dissolved in a suitable solvent, like water or ethanol, to create the precursor solution. Addition of plant extract: The plant extract is then added to the precursor solution and the mixture is heated, stirred for a specific time period.Formation of nanoparticles: The nanoparticles are then formed through the reduction of the metallic ions by the plant extract and are stabilized by the capping agents present in the plant extract solution.

Table 2: Green synthesis routes of ferrite materials

S. N.	Ferrite Name	Plant Used	SynthesisMethod	Reference
1.	Zinc Ferrite	LimoniaAcidissima Juice	Precipitation method	Naik et al. (2019)
2.	Zinc Ferrite	Piper Nigrum	Precipitation method	Din et al. (2020)
3.	Zinc Ferrite	LawsoniaInermis	Precipitation method	Sarala et al. (2020)
4.	Zinc Ferrite	Ginkgo Biloba	Precipitation method	Liaskovska&Tatarchuk (2021)
5.	Zinc Iron Oxide	Moringa Oleifera Extract	Coprecipitation method	Matinise et al. (2018)
6.	Zinc Ferrite	Petroselinum Crispum	Precipitation method	Korotkova *et al.* (2019)
7.	Nickel Ferrite	Clove Extract	Co Precipitation method	Kulkarni *et al.* (2020)
8.	Nickel Ferrite	Terminalia Catappa	Co Precipitation method	Sarala *et al.* (2022)
9.	Nickel Ferrite	Magnifera Indica	Co Precipitation method	Dinkar *et al.* (2021)
10.	Nickel Ferrite	Rosemary	Co Precipitation method	Alijani*et al.* (2020)
11.	Nickel Ferrite	Lime Peel Extract	CoPrecipitation method	Malik *et al.* (2022)
12.	Nickel Ferrite	Pepper Mint	Hydrothernam method	Kalita *et al.* (2022)
13.	Cobalt Ferrite	Okra Extract	Coprecipitation method	Kombaiah*et al.* (2018)
14.	Cobalt Ferrite	Plant Extracts	Precipitation method	GingaŞu*et al.* (2017)
15.	Copper Ferrite	Aloe Vera Extract	Co precipitation	Laokul*et al.* (2011)

5. Applications of spinel ferrite nanoparticles

Because of their distinctive magnetic, electrical, and optical characteristics, ferrite nanoparticles have gainedenough attention lately. They are useful for a lot of applications in many fields because to these characteristics. Following are a few typical uses for ferrite nanomaterials: Ferrite nanoparticles could be used in biomedical engineering for processes including medication administration, magnetic resonance imaging, hyperthermia therapy, and bioseparation. Ferrite nanoparticles are extensively used in magnetic recording medium, including hard disc drives, due to their great magnetic anisotropy, high coercivity, and high magnetic moment. Ferrite nanoparticles have been frequently used in microwave devices such isolators, circulators, and phase shifters because of their unusual magnetic and electrical properties. Ferrite nanoparticles have been employed in environmental remediation processes like water purification and wastewater treatment because of their outstanding adsorption and catalytic properties.

Ferrite nanoparticles have the potential to be employed in energy storage systems like rechargeable batteries and supercapacitors due to their huge surface area, excellent thermal and electrical conductivity, and exceptional electrochemical stability. Ferrite nanoparticles can be used in a range of catalytic processes, including photocatalysis and heterogeneous catalysis, because of their distinctive magnetic and catalytic properties. Magnetic storage methods that heavily use ferrites include hard disc drives, magnetic tapes, and magnetic floppy discs. The synthesized nano-ferritesare ideal materials for magnetic storage applications and an efficient photocatalyst. Because of its distinctive magnetic and dielectric characteristics, ferrite are frequently utilized in microwave device including circulator, isolator and filters. Microwave devices have used nickel zinc ferrite nanoparticles because of their high permeability and low dielectric loss.

Ferrites are used in a wide variety of biomedical processes, including magnetic resonance imaging, targeted medication administration, and hyperthermia therapy. Due to their strong magnetism and biocompatibility, superparamagnetic iron oxide nanoparticles (SPIONs) are frequently utilised in MRI. The removal of heavy metals from contaminated water and the treatment of wastewater are two examples of how ferrites have been employed in environmental applications. Because of their high surface area and superior adsorption capabilities, zinc ferrite

($ZnFe_2O_4 $) nanoparticles have been utilised to remove cadmium (Cd) like ions from water. Because of its large surface area and distinctive magnetic characteristics, ferrites have also been used as catalysts in a variety of chemical processes.

6. Future prospects of ferrite nanomaterials:

Due to its intriguing features and prospective uses in a variety of industries, ferrite nanoparticle research and development have attracted a lot of attention lately. Given that scientists are constantly looking for new ways to create and use ferrite nanoparticles, their future appears to be highly promising. The field of biological applications is one of the key areas where ferrite nanoparticles exhibit strong prospects. Drug administration, magnetic resonance imaging, hyperthermia, and magnetic separation are just a few of the biomedical uses for magnetic nanoparticles. To increase ferrite nanoparticles' effectiveness in various applications, researchers are working to enhance their biocompatibility as well as targeting capabilities.Additionally, ferrite nanoparticles exhibit enormous promise in the fields of environment and energy. They can be utilised in the creation of highperformance energy storage devices like lithium-ion batteries,supercapacitors,etc. Ferrite nanoparticle's distinctive magnetic characteristics can be used in environmental applications such heavy metal ion removal, wastewater remediation etc. Electronics, agriculture sectors, biological applications thus make ferrites promising materials for future.

Conclusion: Green synthesis technology is now in high demand in the scientific research field for nearly all types of nanomaterials. These green methods have been a leading environment-friendly and reduce pollution. Future development of green research will result in the effective and efficient production of desired nanoparticles forms with enhanced characteristics. The application of spinel ferrites in the fields of biomedical sensors, electro-sensors, energy storage devices, biosensors, and data storage systems are all currently the subject of extensive research. As a result, more research is required in spinel ferrite nanoparticles produced through green synthesis.

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