Synthesis of ferrites using various parts of plants: a mini review
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Abstract. Ferrite nanoparticles are magnetic. They also show photocatalytic and antibacterial activities. These unique properties make them extremely useful for various applications e.g., they can potentially be used for electronic equipment, telecommunication systems, radar, sensing material, catalytic and photocatalytic applications, and several biomedical applications. Several methods have been developed to synthesise these materials. These include sol-gel, co-precipitation, ball-milling, combustion, hydrothermal heating, and microwave hydrothermal procedure. Recently, these materials have been using various parts of plants. Hibiscus rosa-sinensis, Hydrangea paniculata, rosemary, nyctanthes arbor-tristis, sesame seed, aegle marmelos, Limonia acidissima juice, aloe vera, amaranthus blitum, ginger and cardamom, were used for this purpose. Ferrites of silver, cobalt, zinc, and nickel were prepared successfully. Few doped ferrites, e.g., silver doped cobalt ferrite, nickel doped zinc ferrite, and zinc doped nickel ferrite were also synthesised. Use of plant-parts evades the necessity of the use of expensive metal salts. Various parts of plants, e.g., flowers, leaves, and seeds act as a gelling agent, chelating agent, reducing agent, and capping agent during reactions. This is a non-toxic, eco-friendly, and cost-effective method. The samples were characterized using various Advance Materials Characterisation Techniques. The results were very much in agreement with the desired values. In a few cases, better antibacterial activities have been observed with ferrites obtained via this method. Left-over parts of food and fruits can be used for this method which may help in waste management. Still, the tremendous scope is left in this area that can be exploited which may include scaling-up of the product.

Keywords: nanoparticles, magnetic materials, chemical and biosynthesis, antibacterial activities

1. Introduction
Particles with size in the range of $10^{-9}$ m are nanoparticles (NPs). Ferrite particles in this size range, are called ferrite NPs (FNPs) [1, 2]. Various forms of FNPs are found in nature as well as synthesised. Spinel ferrites are one of them [3-6]. To our knowledge, all the ferrites synthesised using different parts of plants, are spinels, hence, hereafter, only spinel ferrites are discussed. An overview of spinel ferrite is given in the following section (see sec. 2).

2. Overview of spinel ferrite
Molecular structure, properties, applications, and routes of synthesis of spinels are discussed in this section (see 2.1 to 2.4).

2.1 Molecular structure of spinels [8]
Spinell ferrites have general formula AB$_2$X$_4$, where ‘A’ is a divalent cation, ‘B’ is a trivalent cation, and X is the anion. A list of possible ions is given in Table 1.
Table-1. List of possible ions in AB₂X₄ formula [8].

| Divalent cations (A) | Trivalent cations (B) | Anions (X) |
|---------------------|-----------------------|------------|
| Mg²⁺, Cr³⁺, Fe³⁺, Mn²⁺, Co²⁺, Ni²⁺, Cu²⁺, Zn²⁺, Cd²⁺ | Al¹⁺, Ga¹⁺, In³⁺, Ti³⁺, V³⁺, Cr¹⁺, Mn¹⁺, Fe¹⁺, Co¹⁺, Ni¹⁺ | O, S, Se |

These materials can be categorised as follows,
(i) Normal spinels have general formula AB₂O₄. Here, divalent cations occupy tetrahedral sites whereas trivalent cations occupy octahedral sites. MgAl₂O₄, Mn₃O₄, ZnFe₂O₄, FeCr₂O₄ are normal spinels [8].

(ii) Inverse spinels have the general formula (B(AB)O₄), where all the divalent cations occupy octahedral sites, and the trivalent cations are equally distributed between tetrahedral and octahedral sites. Fe₃O₄, CoFe₂O₄, NiFe₂O₄ are inverse spinels [8].

(iii) Mixed spinels have general formula A₁₋ₓBₓ⁺²[Bₓ₋₁₋ₓAₓ⁻²⁺]O₄²⁻, where x is the degree of inversion. Here, both A and B occupy tetrahedral as well as octahedral sites. Mn-Zn ferrites and MnFe₂O₄ have mixed spinel structures [8].

2.2 Properties of spinel ferrites
These materials show dielectric, magnetic, catalytic, and optical properties [7, 9-11]. Spinel ferrites are paramagnetic. They show saturation magnetisation, and super paramagnetic behaviour [17, 22, 23]. They also exhibit exceptional optical and dielectric properties, for example, they show linear absorption, and photoluminescence emission and nonlinear optical behavior [12, 13]. They also display piezoelectric, pyroelectric, ferroelectric, and antiferroelectric characteristics [9, 14-16].

2.3 Applications of spinels
Spinel ferrites care extremely useful because of their unique characteristics (see sec. 2.2), for example, they can be used for the fabrication of electronic and microwave devices. They can also be used for several biomedical applications, for example, in Medical Resonance Imaging (MRI), in the construction of biosensors, and in inducing magnetic hyperthermia in malignant cells, drug delivery systems, as antibacterial, antiallergic, and antiviral agents. They can be used as catalysts as well, for example, CuFe₂O₄ and AgFe₂O₃ are catalysts [17-30].

2.4 Routes of synthesis of spinels
Efforts have been made to synthesise these materials because of its unique applications. Several methods have been developed for this purpose for example, combustion method [31], precipitation and co-precipitation methods [31], hydrothermal and microwave hydrothermal method [32], sol-gel [33, 34], and green synthesis [35] to name but a few. These materials can also be synthisised using various parts of plants [17-30]. Here, we have reviewed the work related to synthesis of spinel ferrites using various parts of plants in the following sections (see sec 3 to 5).

3. Review of various spinel ferrites obtained via different parts of plants.
The work related to spinel ferrites prepared using various parts of plants are summarised in the following sections [17-30].

3.1 Summary of methods of synthesis and characterisations
Different names were given for these methods by different research groups, for example, green and sustainable process [17], biological green synthesis [18], self-combustion and wet-ferritization [19,
21], self-combustion [20], green synthesis [22, 26, 29], modified sol-gel [23, 30], microwave-assisted green method [24], microwave heating and modified sol-gel [25], chemical co-reduction precipitation and green co-reduction methods [27], and novel hydrothermal method [28]. They used rosemary flower [17], flowers of nyctanthes [18], sesame seed [19], ginger root/cardamom [20], Hibiscus rosasinensis flower/leaves [21, 25], Hydrangea paniculata flower [22], aloe vera leaves [23, 28, 30], Limonium acidissima juice [24], aegle marmelos leaves [26], amaranthus blitum leaves [27], Citrus aurantium flower [29] to synthesise NiFe$_2$O$_4$, ZnFe$_2$O$_4$, CoFe$_2$O$_4$, Ag-doped CoFe$_2$O$_4$, CuFe$_2$O$_4$, AgFeO$_2$, ferrites of manganese and magnesium, and Ni-Zn ferrites. In most of the cases metal nitrates and chlorides were taken as source of metal ions. Authors claimed the roles of different parts of plants in the reaction system as reducing agents [19, 26, 30], capping agents [19, 29], stabilizing agents [19], chelating agents [19], gelling agents [19], and coating agents [29]. Remarkably, the samples were obtained successfully in acidic as well as in alkaline mediums. The products were calcined at high temperatures and were characterised using Advanced Materials’ Characterization Techniques viz., X-ray Diffraction Techniques (XRD) was used for the study of crystalline structures, Fourier Transform Infrared Spectroscopy (FTIR) was used for the compositional analysis of the samples (this shows functional groups present in the molecules of the sample), Vibrating Sample Magnetometer (VSM) was used for the study of magnetic behaviour, Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) was used to analyse the morphology of the samples whereas Energy Dispersive X-ray Analysis (EDX/EDAX) using Field Emission Scanning Electron Microscopy (FESEM) was performed for the elemental analysis of the samples [17-30]. Ultraviolet-Visible (UV-Vis) was used to confirm the formation of the ferrites [19, 22, 24-27, 29]. In the following sections we have reviewed the results obtained in these works (see sec. 3.2 to 3.7).

3.2 XRD for crystal structure study

Crystalline structures of samples were studied using X-ray diffraction (XRD) [17-30]. The XRD patterns of ZnFe$_2$O$_4$ show broad peaks which suggested the creation of single-phase spinel structure. Balasubramanian et al. reported that the increase in calcination temperature improved the crystallinity of the sample as XRD data showed reduction in the width of the XRD-peak [18]. Preparation of CoFe$_2$O$_4$ via self-combustion method using the aqueous extracts of ginger root and Hibiscus rosasinensis, demonstrated the formation of cubic spinel structures of the samples. Whereas single phase spinel CoFe$_2$O$_4$ were obtained from aqueous extracts of cardamom seeds invariably [19-21], CuFe$_2$O$_4$ NPs were also obtained in single-phase cubic spinel structures [25]. Few important intensities in $\theta/2\theta$ as obtained by XRD is listed in Table 2.

| NiFe$_2$O$_4$ | ZnFe$_2$O$_4$ | AgFeO$_2$ |
|----------------|----------------|----------------|
| **Intensity(º/2θ)** | **Assign.** | **Observation** | **Ref.** | **Intensity(º/2θ)** | **Assign.** | **Observation** | **Ref.** | **Intensity(º/2θ)** | **Assign.** | **Observation** | **Ref.** |
| 30.02 | (220) | Spinel | [17] | 29.72 | (220) | Cubic structure | [26] |  |
| 35.67 | (311) | Spinel | [17] | 35.08 | (311) | Cubic structure | [26] |  |
| 38.19 | (222) | Spinel | [17] | 36.60 | (222) | Cubic structure | [26] |  |
| 54.01 | (422) | Spinel | [17] | 42.72 | (400) | Cubic structure | [26] |  |
| 57.98 | (511) | Spinel | [17] | 53.05 | (422) | Cubic structure | [26] |  |
| 62.92 | (440) | Spinel | [17] | 56.41 | (511) | Cubic structure | [26] |  |
| 27 | (111) | Cubic crystalline (fcc) | [22] | 62.07 | (440) | Cubic structure | [26] |  |
| 45 | (220) | Cubic crystalline (fcc) | [22] | 70.21 | (620) | Cubic structure | [26] |  |
| 54 | (311) | Cubic crystalline (fcc) | [22] | 74.37 | (533) | Cubic structure | [26] |  |
| 66 | (400) | Cubic crystalline (fcc) | [22] | 78.40 | (444) | Cubic structure | [26] |  |
| 85 | (422) | Cubic crystalline (fcc) | [22] |  |
| 91 | (511) | Cubic crystalline (fcc) | [22] |  |
| 101 | (440) | Cubic crystalline (fcc) | [22] |  |
| 129 | (533) | Cubic crystalline (fcc) | [22] |  |
| 136 | (622) | Cubic crystalline (fcc) | [22] |  |
|  |  |  |  |  |  |  |

Table 2. List of crystalline peaks obtained for some important ferrites.
3.3 UV-Vis for the confirmation ferrite formation

In this technique visible light is used to obtain the spectrum [36]. Zinc ferrite (ZnFe₂O₄) NPs absorbs visible light, which produces a single reflection band within the range of (200–1000) nm which is suitable for photo catalysis [18]. The maximum absorption peak at 355 nm demonstrated the formation of NiFe₂O₄ NPs [22]. ZnFe₂O₄ NPs obtained via microwave-assisted green synthesis produced a luminescence spectrum at 360 nm [24]. The broad absorbance peak at 500 nm indicates that the ZnFe₂O₄ with spinel structures were obtained when synthesised using hydrothermal method [26].

3.4 FTIR for compositional analysis

Invariably all the research groups used FTIR analysis to study the compositions of the samples. The assignments of bands are listed in Table-3 for various ferrites and are in good agreement with the literature for all ferrites synthesised by using different parts of plants. FTIR data confirmed the functional groups present in the spinel ferrites [17-30]. Few important functional groups obtained by several research groups with absorption band-positions are listed in Table 3.

Table 3. List of functional groups obtained at various absorption bands in FTIR analysis.

| ZnFe₂O₄ | CoFe₂O₄ | NiFe₂O₄ |
|---------|---------|---------|
| Intensity(º2θ) | Assign. | Observation | Ref. | Intensity(º2θ) | Assign. | Observation | Ref. | Intensity(º2θ) | Assign. | Observation | Ref. |
| 34.8 (101) | Crystalline nature | [27] | 37.07 (102) | Crystalline nature | [27] | 40.63 (103) | Crystalline nature | [27] | 43.78 (006) | Crystalline nature | [27] |
| 18.170 (111) | Single phase cubic | [24] | 50.52 (105) | Crystalline nature | [27] |
| 29.940 (220) | Single phase cubic | [24] | 56.89 (106) | Crystalline nature | [27] |
| 35.250 (311) | Single phase cubic | [24] | 59.64 (008) | Crystalline nature | [27] |
| 42.870 (400) | Single phase cubic | [24] | 60.9 (110) | Crystalline nature | [27] |
| 53.170 (422) | Single phase cubic | [24] | 62.97 (112) | Crystalline nature | [27] |
| 56.660 (511) | Single phase cubic | [24] | 63.25 (107) | Crystalline nature | [27] |
| 62.220 (440) | Single phase cubic | [24] | 68.77 (114) | Crystalline nature | [27] |
| 70.620 (620) | Single phase cubic | [24] | 72.09 (201) | Crystalline nature | [27] |
| 73.600 (533) | Single phase cubic | [24] | | | |

| NiFe₂O₄ | ZnFe₂O₄ |
|---------|---------|
| Abs. (cm⁻¹) | Assign. | Ref. | Abs. (cm⁻¹) | Assign. | Ref. |
| 2570 -CH | [22] | 605 Zn-O tetrahedral | [24] |
| 1980 -C=H | [22] | 405 Fe-O octahedral | [24] |
| 1500 -C=O | [22] | 2923 & 1393 C-H stretch | [24] |
| 1346 -Fe-O | [22] | 3439 & 1622 H-O-H | [24] |
| 1062 -Ni-O | [22] | 114 CO₂ | [24] |

| ZnFe₂O₄ | ZnFe₂O₄ |
|---------|---------|
| Abs. (cm⁻¹) | Assign. | Ref. | Abs. (cm⁻¹) | Assign. | Ref. |
| 3382 (m, b)⁵ OH/H₂O | [19] | 3448.92 OH | [17] |
| 3007 (w)² C-H aromatic | [19] | 400-700 C-H bending out of plane | [17] |
| 2925 (vs)³ CH₃ asymmetric | [19] | 1637.72 C-C benzoid ring | [17] |
| 2853 (s)⁴ CH₂ symmetric | [19] | 600 M-O intrinsic stretch (tetrahedral) | [17] |
| 575, 570, 587 Fe-O stretch (tetrahedral) | [20] | 450 M-O, stretch (octahedral) | [17] |
| 418 Fe-O & Co-O octahedral | [20] | 3385.52 OHH bending | [17] |
| 591 & 386 M-O spinel | [21] | 1604.41 H-OH stretch (carboxylic acid) | [17] |
| 2942 Phenol | [21] | > 600 Fe-O stretch (tetrahedral) | [17] |
| 3564, 3271 OH | [21] | < 400 Ni-O stretch (octahedral) | [17] |
| 587 Fe-O | [21] | | |

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CuFe₂O₄  

| Abs (cm⁻¹) | Assign. | Ref. | Abs (cm⁻¹) | Assign. | Ref. |
|------------|---------|-----|------------|---------|-----|
| 3400-3200  | -OH stretch | [26] | 3364       | OH       | [29] |
| 1633       | -C≡C-      | [26] | 1620       | -C=C- aromatic | [29] |
| 1322       | Methyl group | [26] | 1053       | C=O–C etheric | [29] |
| 1038       | C=O-C      | [26] | 563        | Fe-O     | [29] |

CuFe₂O₄

| Abs (cm⁻¹) | Assign. | Ref. |
|------------|---------|-----|
| 3400-3200  | -OH stretch | [26] |
| 1633       | -C≡C-      | [26] |
| 1322       | Methyl group | [26] |
| 1038       | C=O-C      | [26] |

Note: 1. m = medium, b = broad; 2. w = weak; 3. vs = very strong; 4. s = strong

3.5 VSM for magnetic behaviour

The magnetic properties of ferrites nanoparticles were studied by using Vibrating Sample Magnetometer (VSM) technique. Green synthesised nickel ferrite (NiFe₂O₄) NPs show reduction in saturation magnetisation due to disorder in surface spin, creation of thin oxide layer, and size detection [17]. Cobalt ferrites (CoFe₂O₄) NPs made via wet ferritization method, showed that the coercivity and remanence values increases with heat treatment, because the particle size increases. It changes from superparamagnetism monodomain to the multi-domain sizes. As a result, the soft magnetic materials change to hard magnetic materials [19]. It is observed that saturation magnetisation value of Ag doped CoFe₂O₄ NPs synthesised via self-combustion method using leaves and flowers of *Hibiscus rosasinensis* extract showed a change in magnetic behaviour when heat treated as heat treatment increased the particle sizes [21]. NiFe₂O₄ NPs prepared using *Hydrangea paniculata* flower extract showed soft magnetic behaviour [22]. Copper ferrite (CuFe₂O₄) samples show ferrimagnetic behaviour. Its saturation magnetization value increases with rise in calcination temperature [23]. Zinc ferrite (ZnFe₂O₄) NPs show soft magnetic behaviour which was demonstrated by thin hysteresis loop [24]. Gummadi et. al. reported that the magnetic behaviour of silver ferrite (AgFe₂O₄) NPs depends upon the routes of synthesis [27]. According to VSM analysis CoFe₂O₄, MgFe₂O₄, and MnFe₂O₄ are soft magnets whereas ZnFe₂O₄ and NiFe₂O₄ are superparamagnetic in nature [28]. Hysteresis loops of CoFe₂O₄ prepared using extracts of sesame seeds as obtained using VSM is depicted in fig. 1. Note that permission has been taken from the journal.
Figure 1. Hysteresis loop showing the difference in magnetic behaviour of samples prepared at different conditions. Three parts of the figure can be explained as follows:

1a. samples prepared via self-combustion method followed by calcination at 800 °C for 1 hr.
1b. samples prepared via wet ferritization method.
1c. samples prepared via wet ferritization method followed by calcination at 800 °C for 1 hr [19].

3.6 Electron microscopy
The morphology and microstructure of the samples were analysed by using Scanning Electron Microscopy (SEM, see 3.6.1), Transmission Electron Microscopy (TEM, (see 3.6.2)), and High-Resolution Transmission Electron Microscopy (HRTEM (3.6.2)). The compositions of materials were analysed through Energy Dispersive X-ray Analysis (EDAX, 3.6.3) by using Field Emission Scanning Electron Microscopy (FESEM).

3.6.1 Study of morphology of ferrite NPs using SEM:
CoFe$_2$O$_4$ NPs with uniform morphology were obtained with aqueous extracts of sesame seeds, whereas agglomerated particles were achieved with ginger root and cardamom seeds [19, 20]. NiFe$_2$O$_4$ NPs are formed as fine and soft agglomerated clusters from Hydrangea paniculata flower extract [22] whereas ZnFe$_2$O$_4$ NPs show uniform size distribution when synthesised using Limonia acidissima juice [24]. SEM results showed that AgFeO$_2$ are less agglomerated with smooth morphology [27]. The morphology of MFe$_2$O$_4$, where M = Ni, Co, Mn, Mg, Zn for different samples shows cluster of particles [28]. Images of CoFe$_2$O$_4$ prepared using extracts of sesame seeds as obtained using FESEM is depicted in fig. 2 (‘a’ to ‘e’). Note that permission has been taken from the journal.
Figures. 2 Images of CoFe₂O₄ NPs as observed under FESEM synthesised using extracts of sesame seeds. Five views have been taken by the authors. Details of the images can be seen in the work published by Mindru et. al. [19].

3.6.2 Study of morphology of ferrite NPs using TEM and HRTEM studies

HRTEM study of NiFe₂O₄ NPs (synthesised using rosemary) showed the formation of rod-like nanostructure [17] whereas spherical NPs were obtained when synthesised using Hydrangea paniculata flower extract [22]. Note that TEM was used to study the morphology of the particles in the latter case (i.e., ref. 22). TEM micrographs of ZnFe₂O₄ NPs show uniform spherical shape when synthesised using Limonia acidissima juice [24], whereas polyphenol coated ZnFe₂O₄ NPs were obtained when synthesised using citrus aurantium [29]. Ni-Zn ferrites NPs with spherical shape were obtained when synthesised using aloe-vera. Interestingly, particle size increases with increase in nickel concentration [30].

3.6.3 Compositional analysis by performing EDX analysis using FESEM.

Energy Dispersive X-ray Analysis (EDX/EDAX) was used to identify chemical compositions of the samples. For example, the chemical composition of NiFe₂O₄ NPs confirmed the presence of Fe, Ni, and O elements in the samples [17]. This technique can also be used to detect trace elements for example, CoFe₂O₄ contained traces of elements like Si, C, P and S [21].

Figure 3a Images of NiFe₂O₄ (bimetallic) nanorods as observed under HRTEM, 3b EDS results and 3c SAED patterns of the samples synthesised using Rosemary [17].

3.7 Role of parts of plants in the synthesis of ferrites.

Use of different parts of plants evades the need of expensive and not very environmentally friendly metal salts in the preparation of ferrites. The group of chemicals released in the reaction system acted as capping agents, reducing agents, stabilising agents, and chelating agents [17-30]. Mindru et. al. reported that the plant extracts contained several metabolites e.g., carbohydrates, phenols,
polysaccharides, vitamins, and amino acids. These chemicals also provided energy to the reaction system which was termed as ‘fuel’ by the authors [19]. Jangjau et. al. believed that the chemicals from plant extract behaved as coating agents apart from reducing and capping agents [29] whereas Maensiri et. al. believed that plant extracts were present only as reducing agent in the reaction system as well [23].

4. Summary
Ferrite NPs have numerous unique properties enabling them for several applications ranging from electronics to biomedical applications. They can also be used as catalysts. Hence, several methods have been developed to synthesise these materials. Recently, few research groups started using various parts of the plants to synthesise this group of materials. Use of various parts of plants is cost effective and more environmentally friendly as it evades the use of metal salts. This method does not require the application of remarkably high temperatures or requirement of extremely sophisticated laboratory infrastructure which were added advantages. The results obtained using various Advanced Materials Characterization Techniques agreed with the existing literature. XRD studies showed the formation of crystals whereas FTIR studies showed the presence of various functional groups e.g., O-H, M-O, C-H, C-C, C=O, and H-O-H, to name but a few. Chemical compositions were also studied using EDAX analysis. SEM and TEM studies showed that the particles with smooth morphology were formed in various cases. Magnetic properties were studied using VSM. It was observed that the magnetic behaviour was dependent upon the size of the particles in several cases which in turn was dependent upon the routes of synthesis e.g., calcination temperature. This demonstrated that ferrite NPs with desired magnetic can be produced by modifying the routes of synthesis.

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