REVIEW



Plant-Mediated Synthesis and Applications of Iron Nanoparticles

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Abstract

Nanoscale iron particles have attracted substantial interest due to their unique physical and chemical properties. Over the years, various physical and chemical methods have been developed to synthesize these nanostructures which are usually expensive and potentially harmful to human health and the environment. Synthesis of iron nanoparticles (INPs) by using plant extract is now of great interest in order to develop a novel and sustainable approach toward green chemistry. In this method the chemical compounds and organic solvents are replaced with phytochemicals and aqueous matrixes, respectively. Similar to any chemical and biochemical reaction, factors such as reaction temperature, concentration of iron precursor, concentration of leaf extract, and reaction time have critical effects on the reaction yield. This review focuses on the novel approaches used for green synthesis of INPs by using plant resources. The currently available statistics including the factors affecting the synthesis process and potential applications of the fabricated nanoparticles are discussed. Recommendations are also given for areas of future research in order to improve the production process.

Keywords Biosynthesis · Eco-friendly · Environmental friendly · Green chemistry · Green synthesis · Metal nanoparticles

Introduction

Recently, nano- and microstructures have gained applications in various science and technologies [1–8]. Iron nanoparticles (INPs) are among the most interesting novel materials [3] due to their unique physicochemical properties,

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high catalytic activity, high magnetism, low toxicity, and microwave absorption ability [9, 10]. INPs can be classified into three major groups namely (1) iron oxide nanoparticles (IONs) (i.e., magnetite; Fe₃O₄, hematite: α -Fe₂O₃, maghemite; γ -Fe₂O₃), (2) iron oxide hydroxide (FeOOH) nanoparticles, and (3) zero-valent iron (ZVI) nanoparticles [11–14]. These particles cover a wide range of applications including in drug delivery [14], magnetic targeting [15], hyperthermia [16], thermal-ablation [17], stem cell sorting and manipulation [18], gene therapy [19], negative MRI contrast enhancement [20], food preservation [21], bioprocess intensification [22, 23], antimicrobial agents [24], bioseparation [25], ferrofluids [26], environmental remediation [8, 27], lithium-ion batteries [28], and pigments [29].

To date, different physical and chemical processes have been developed for the production of INPs. These processes fall into two general techniques including top-down and bottom-up methods as shown in Fig. 1. In the top-down methods, nanoscale structures are gradually obtained from bulk materials by employing several techniques including etching, laser ablation, mechanical milling and sputtering. Although these approaches may successfully produce pure and good-specified nanoparticles, they are not economically sustainable and usually require extensive use of equipment



Fig.1 Top-down and bottom-up approaches for the preparation of $\ensuremath{\mathrm{INPs}}$

[30, 31]. In the bottom-up approaches, metal precursors (i.e., metal ions) are used in solid, liquid, or gas phases to produce nanoparticles. These procedures can be conducted in aerosol process, atomic condensation, sol–gel phases, vapor deposition, thermal decomposition, co-precipitation, and via green synthesis [32].

To date, many researchers have reported synthesis of INPs by using biological systems, including plants [33], algae, and different microorganism (yeast, fungi, diatoms, and bacteria) [3, 34, 35]. Among the developed biological systems, plant-mediated synthesis attracted a significant scientific interest [36]. However, use of live plants for producing INPs has major drawbacks such as heterogeneity in size and shape of the produced nanoparticles due to biosynthesis in different organs and various tissues. On the other hand, isolation of INPs from plant body is a hard and tedious procedure which subsequently results in low recovery and purity [3]. Green synthesis of INPs using plant extract addresses aforementioned problems as it is a fast, simple, and easy to scale-up for industrial productions [37]. Since now extracts of various parts of plants such as fruit extract, seed powder extract, seed exudate, peel extract, bran extract, bark extract, flower extract, and mostly leaf extract have been used for green synthesis of nanoparticles [38–55]. The extract can act

as a natural source for reducing and capping agent in a one pot synthesis reaction which eliminates multistep synthesis practice problems and costs of chemical reagents [6, 56]. Scientists usually try to use extracts from nonfood sources, such as grasses and trees, as a feedstock for industrial green synthesis reactions to reduce the production costs. Also, in order to increase the techno-economic opportunities, green synthesis reactions are usually done in ambient temperature and atmosphere [54–58]. While, chemical synthesis of INPs is usually done at high temperatures and under protected atmosphere [2, 7, 15, 22, 24, 59–62]. These opportunities can make the plant-mediated green synthesis one of the top economic approaches for large-scale synthesis of nanoparticles. In the current review we explain the potential of plant extracts for green synthesis of various INPs and the applications of the prepared nanoparticles.

Green Synthesis of Iron Nanoparticles Using Plant Extract

In this production technique, the extract usually contains a mixture of plant extract and one of the various iron salts [e.g., FeCl₃·6H₂O, FeCl₂·4H₂O, FeSO₄ or Fe(NO₃)₃] as iron precursor. The reaction usually occurs spontaneously at room temperature under ambient air. Reaction rate is fast and generally completes within a few minutes. Phytochemicals in plant extract simply convert iron ions to INPs. These biomolecules include a variety of water soluble metabolites (e.g., polyphenols, sugars, alkaloids, phenolic acids, and proteins) and coenzymes that can act as reducing and stabilizing agents in the biosynthesis reaction (Fig. 2) [63–65]. The physicochemical characteristics of the synthesized INPs by using different plant extracts are not the same (Table 1). Reaction conditions (i.e., temperature, concentration of iron precursor, amount of plant extract, and time) and chemical specifications of the plant extract (i.e., preparation temperature, concentration, pH, and phytochemical molecules) have significant effects on the physicochemical properties of the resultant nanoparticles [65-68]. Various INPs including zero valent (ZVI), ferrous ferric oxide (Fe₃O₄), ferric oxide (Fe₂O₃), iron oxide hydroxide (FeOOH), and iron mineral complex nanoparticles can be fabricated by using this synthesis approach. Each of these INPs has unique properties that make them potentially suitable for specific scientific and technical applications (Fig. 3).

Extracellular plant-mediated synthesis of INPs is a single-step method, which involves the plant extract preparation and mixing it into aqueous iron ion solutions in controlled condition [69, 70]. In regard to prepare plant extract, harvested leaves were rinsed with deionized water to remove possible mud and dust, and were dried at room temperature. Leaf aqueous extract can be prepared by



Fig. 2 Schematic illustration of the plant-mediated synthesis of INPs

boiling dried leaves in deionized water. Dried leaves and water are usually used in a ratio of 2–10:100, respectively. The boiling should be done under reflux to prevent evaporation. After cooling at room temperature, the solution can be separated by filtration and leaf microparticles can be excluded by centrifugation [71–76]. Plant metabolites such as proteins, amino acids, vitamins, polyphenols, and caffeine could act as green bioreductant and stabilizing agents for the production of INPs. In summary, iron ions bind to these metabolites and reduced to iron atoms and the formation of iron nucleus occurs. Iron nucleuses subsequently

grow to the stable INPs [69, 70, 77]. This reaction usually completes under vigorous stirring at room temperature.

Zero-Valent Iron Nanoparticles

Zero oxidation state of iron with incompletely filled d-orbitals is called ZVI, which has the electron donor activity making it highly reactive. ZVI has been recognized as a great electron donor without taking its particle size and has long been identified for its remediation applications in various environmental and industrial processes [78]. In general, for fabrication of metal nanoparticles in the zero-valent state by bottom-up approach, we need to reduce metal ions to atoms. Atoms can attach to each other by metallic forces to produce the nanoparticles. Phytochemicals in the plant extract were reported to be capable of doing this job and also stabilizing the nanoparticles by capping them (Fig. 4) [79]. For example, Hoag et al. (2009) reported the synthesis of spherical and highly small size ZVI nanoparticles (5–15 nm) by exposing the aqueous green tea leaf extract to ferric nitrate solution. The rate of nanoparticles synthesis was extremely high during the spontaneous reaction at room temperature. Prepared nanoparticles were effective in degradation and removal of organic contaminations such as bromothymol blue [80].

Pattanayak and Nayak generated the stable ZVI nanoparticles by using the leaf extracts of Azadirachta indica (Neem) [81]. Machado et al. (2013) carried out an experiment by using the leaf extracts of different tree species (apple, apricot, avocado, cherry, eucalyptus, kiwi, lemon, mandarin, medlar, mulberry, oak, olive, orange, passion fruit, peach, pear, pine, pomegranate, plum, quince, raspberry, strawberry, black tea, green tea, vine, and walnut). The antioxidant capacity of these samples was measured by "ferric reducing antioxidant power" (FRAP) method. The smallest ZVI nanoparticles (5–10 nm) were formed by using mulberry and pomegranate leaf extracts, while ZVI nanoparticles produced by medlar and vine leaf extracts were found to be more effective for environmental remediation [82]. Huang et al. (2014) compared the capability of three plant extracts (green tea, oolong tea, and black tea) for the formation of ZVI nanoparticles. The authors reported that green tea extract could produce more ZVI nanoparticles as compared to the oolong and black tea extract. The produced nanoparticles varied in aggregation state and size diversity leading to formation of various catalysts for the degradation of malachite green [77]. Several studies have shown that the green tea extract contains polyphenols and caffeine, which play a key role as reducing and stabilizing agents during the formation of INPs. For instance, Xiao et al. (2016) reported that INPs synthesized by S. jambos and Alston extract exhibited better removal capacity for chromium [83]. Elsewhere in a

Table 1 Various plants which have been used for the green synthesis of INPs and characteristic features of the resulted particles

Plant	Used part	Iron precursor	Resulted INPs	Size (nm)	Shape	References
Hordeum vulgare Rumex acetosa	Leaves	FeCl ₃ .6H ₂ O	Magnetite	Up to 30 10–40	Spherical	[70]
Green tea	Leaves	FeCl ₂ .4H ₂ O	Magnetite FeOOH	40–60 nm	Irregular cluster	[98]
Eucalyptus	Leaves	FeSO ₄ .7H ₂ O	IONPs ZVI NPs	20–80 nm	Spherical	[73]
Sorghum spp	Powder	FeCl ₃ .6H ₂ O	FeOOH	50 nm	Irregular cluster	[<mark>63</mark>]
Tangerine	Peel	FeCl ₂ .4H ₂ O	IONPs	50–200 nm	Spherical	[<mark>67</mark>]
Soya bean	Sprouts	FeCl ₃ .6H ₂ O	Magnetite	8 nm	Spherical	[138]
Watermelon	Rinds	FeCl ₃ .6H ₂ O	Magnetite	20 nm	Spherical	[72]
Mimosa pudica	Root	FeSO ₄ .7H ₂ O	Magnetite	60–80 nm	Spherical	[101]
Caricaya papaya	Leaves	FeCl ₃ ·6H ₂ O	Magnetite	Irregular	Irregular	[<mark>68</mark>]
Green tea Oolong tea Black tea	Leaves	FeSO ₄ ·7H ₂ O	ZVI, IONPs, FeOOH, and Fe_2O_3	40–50 nm	Spherical	[77]
Eucalyptus Green tea	Leaves	FeSO ₄ ·7H ₂ O	ZVI, FeOOH	20–80 nm	Quasi-spherical	[120]
Omani mango	Leaves	FeSO ₄ ·7H ₂ O	Maghemite Hematite	15 ± 2	Nanorods	[105]
Salvia officinalis	Leaves	FeCl ₃	Fe ₂ O ₃	5–25 nm	Spherical	[<mark>96</mark>]
Eucalyptus	Leaves	FeSO ₄ ·7H ₂ O		60–20 nm		[89]
Pomegranate Mulberry Cherry	Leaves	FeCl ₃ ·6H ₂ O	ZVI	10–30 nm	Spherical	[82]
Mulberry Pomegranate Peach Pear Vine	Leaves	FeSO ₄ ·7H ₂ O	ZVI	5–10 nm 100 nm	Spherical irregular	[64]
Castanea sativa Eucalyptus globulus Ulex europaeus Pinus pinaster	Leaves	Fe(NO ₃) ₃ ·9H ₂ O	Maghemite			[91]
Sapindus mukorossi	Leaves	Ferric nitrate Ferric chloride	α-FeOOH α-Fe ₂ O ₃ β-FeOH	50 nm	Rod-like	[65]
Eucalyptus	Leaves	FeCl ₃ ·6H ₂ O	IONPs	80 nm	Spherical	[103]
Black tea	Leaves	Ferrous sulfate	FeOOH Fe ₂ O ₃	40–50 nm	Round	[71]
Mansoa alliacea	Leaves	FeSO ₄ ·7H ₂ O	β-Fe ₂ O ₃	18 nm		[74]
Andean blackberry	Leaves	FeSO ₄ ·7H ₂ O	Magnetite	40–70 nm	Spherical	[102]
Amaranthus spinosus	Leaves	FeCl ₃	IONPs	58–530 nm	Spherical	[104]
Eucalyptus	Leaves	FeCl ₃	Fe-p NPs	40–60 nm	Cubic	[<mark>97</mark>]
Green tea	Leaves	Fe(NO ₃) ₃ ·9H ₂ O	Fe-p NPs	70 nm	Spherical	[75]
Eucalyptus tereticornis Melaleuca nesophila Rosmari- nus officinalis	Leaves	FeCl ₃	Fe–p NPs	50–80 nm	Spherical	[95]
Shirazi thyme Pistachio green	Leaves	FeSO ₄ ·7H ₂ O	ZVI AND IONPs FeOOH	40–70 nm	Spherical	[76]
Coffee Green tea Parthenocissus tricuspidata	Leaves	FeCl ₃ ·6H ₂ O	ZVI	19.6 ± 25.8 nm	Triangular	[37]
Carob tree	Leaves	FeCl ₃ ·6H ₂ O	Magnetite	5–8 nm		[106]
Passiflora tripartite	Leaves	FeCl ₃ ·6H ₂ O	Magnetite	22.3 ± 3 nm.	Spherical	[99]

Plant	Used part	Iron precursor	Resulted INPs	Size (nm)	Shape	References
Orange Lime Lemon Mandarin	Pulp Peel Albedo		ZVI	3–300 nm	Spherical Cylindrical Irregular	[84]
Oolong tea	Leaves	FeSO ₄ ·7H ₂ O	ZVI, Fe ₃ O ₄ FeOOH Fe ₂ O ₃	40–50 nm	Spherical	[94]
Camellia sinensis Syzygium aromaticum Mentha spicata Punica granatum juice Red wine	Leaves Fruit	FeCl ₃ ·6H ₂ O	FeNPs	60 nm	Spherical	[121]
Lawsonia inermis Gardenia jasminoides	Leaves	FeSO ₄ ·7H ₂ O	FeNPs	21–32 nm		[125]
Urtica dioica	Leaves	FeCl ₃ ·6H ₂ O	ZVI	21–71 nm	Spherical	[69]
Rosa damascene Thymus vulgaris Urtica dioica	Leaves	FeCl ₂ ·4H ₂ O	ZVI	100 nm		[100]

Fig. 3 Applications and most important properties of different green synthesized INPs



separate study, Machado et al. (2014) used the extracts of fruit waste fractions (peel, albedo, and pulp) of orange, lime, lemon, and mandarin for the reduction in ferric ions. Their results have shown that the extracts of the fruit fractions have produced ZVI nanoparticles with different sizes, shape, and reactivity [84]. Similarly green synthesis of ZVI nanoparticles was also reported by using *Zataria multiflora, pistachio green* hulls [76], coffee, *Virginia creeper* (*Parthenocissus tricuspidata*) [37], *Amaranthus dubius* leaf extract [66], and Eucalyptus leaf extracts [73].

Magnetite (Fe₃O₄) Nanoparticles

Magnetite nanoparticles exhibit unique and useful magnetic properties which makes them an ideal candidate for biotechnological, biomedical [24, 60], and in environmental remediation [8]. The size distributions, shape, and biocompatibility of the Fe₃O₄ particles are of paramount importance for medical application [85–87]. The extract of various plants can produce magnetite nanoparticles from an aqueous iron ion solution. Aqueous extracts of *Hordeum vulgare* (a



Fig. 4 Schematic illustration for the formation and stabilization of ZVI nanoparticles

monocotyledonous plant) and *Rumex acetosa* (a dicotyledonous plant) were used for the synthesis of magnetite nanoparticles (Makarov et al. 2014b). The synthesized INPs by using *H. vulgare* leaf extract were colloidally unstable and were prone to aggregation. However, particles produced by second plant extracts were highly stable. The authors suggested that the higher stability of INPs synthesized by *R. acetosa* extracts was due to the presence of organic acids (such oxalic or citric acids) as stabilizer agent on the surface of INPs [70].

Green synthesis of magnetite nanoparticles was demonstrated by reduction in iron ions (FeSO₄) with aqueous green tea extracts, oolong tea extracts, and black tea extracts (Kuang et al., 2013). The authors fabricated Fe⁰, FeOOH, and maghemite nanoparticles in addition to the magnetite nanoparticle [88]. Elsewhere, Zhuang et al. (2015) reported the use of aqueous extract of Eucalyptus leaf in the extracellular synthesis of magnetite nanoparticles through investigation on the effect of β -cyclodextrin (β CD) on the stability of IONPs. The authors found that adding β CD caused the water solubility of iron particles due to their host–guest interaction [89]. Successful demonstration of Fe₃O₄ nanoparticles synthesis on reduction in ferric chloride solution by aqueous extract of *Caricaya papaya* leaves at room temperature was also recently reported [68].

Iron (III) Oxide (Fe₂O₃) Nanoparticles

Iron (III) oxides include more than one crystal structure that shows different structural and magnetic properties. There are four types of these minerals including α -Fe₂O₃ (hematite), γ -Fe₂O₃ (maghemite), β -Fe₂O₃, and ϵ -Fe₂O₃ [90]. Martínez-Cabanas et al. (2016) reported the successful synthesis of maghemite nanoparticles by using Chestnut tree (*Castanea sativa*) extracts, Eucalyptus (*Eucalyptus globulus*) extracts, Gorse (*Ulex europaeus*) extracts, and Pine (*Pinus pinaster*) extracts. In their study the authors selected *E. globulus* as the best choice for green synthesis of iron (III) oxide nanoparticles [91]. Ali et al. (2016) reported the use of black tea leaf extract for the synthesis of Fe₂O₃ nanoparticles, while iron (III) oxide nanoparticles were successfully synthesized by using aqueous extract of green, black, and oolong tea [77, 88].

Iron Oxyhydroxides (FeOOH) Nanoparticles

Iron oxyhydroxides consist of arrangement of Fe cations and OH⁻ and O²⁻ anions. These materials possess different polymorphic forms including goethite (α -FeOOH), akaganéite (β -FeOOH), lepidocrocite (γ -FeOOH), feroxyhyte $(\delta$ -FeOOH). Iron oxyhydroxides are widespread in nature and are of great industrial importance. Goethite and akaganéite possess superior lithium storage properties and high capacities to be used as electrodes in lithium-ion batteries [92]. FeOOH nanoparticles can be chemically synthesized from Fe(III) or Fe(II) in aqueous solutions via precipitation or oxidation/precipitation reaction [93]. However, in recent year, several green approaches have been developed for the synthesis of FeOOH nanoparticles. For instance, extract of black tea leaves was used for the synthesis of iron oxyhydroxides nanoparticles [71], while Huang et al. (2014) reported the use of oolong tea leaves extracts as a reducing agent for formation of iron oxyhydroxides nanoparticles [94]. Iron oxyhydroxides nanoparticles were synthesized for the reduction in iron (II) sulfate solution by aqueous extract of green, black, and oolong tea [88]. The utilization of sorghum bran extract for the synthesis of FeOOH nanoparticles has also been reported in literature [63].

Iron Mineral Complex Nanoparticles

In this type of nanomaterials ferric ion is located in globular nanoparticles chelated by organic material such as polyphenols [14, 95, 96]. Wang et al. (2013) for the first time reported the formation of iron-polyphenol complex nanoparticles, where the authors used Eucalyptus leaves extract to produce Fe–polyphenol nanoparticles in aqueous solution at ambient conditions. Redox potential of Eucalyptus leaves extracts was measured by the cyclic voltammogram and showed a broad peak at + 0.4 V implying that Fe³⁺ can be reduced to Fe²⁺ by the aqueous leaves extract. However, reduction of Fe²⁺ to ZVI by the leaves extract is a difficult process. Therefore, Fe²⁺–polyphenol complexes were formed in a process that is commonly referred to as autoxidation [96–98].

Wang et al. (2014) reported the use of the extracts of *Eucalyptus tereticornis, Melaleuca nesophila*, and *Rosmarinus officinalis* leaves as a reducing and capping agent for production of iron–polyphenol nanoparticles. The synthesized Fe–polyphenol nanoparticles were utilized for decolorization of acid black 194 [95]. Markova et al. (2014) demonstrated the synthesis of Fe–polyphenol nanoparticles by using aqueous extract of green tea and found that the fabricated nanoparticles were toxic against different aquatic organisms [75]. In addition, Fe–polyphenol nanoparticles were successfully synthesized using the aqueous extract of Sage (*Salvia officinalis*) leaves [96].

Semi-green Synthesis of Iron Nanoparticles

In this technique, which is regarded as partially green method, nanoparticles are produced with combination of chemical and biological reducing agents, so it is less toxic and more cost-effective as compared to the chemical synthesis routes [37]. Several authors have reported the formation of iron nanoparticle via semi-green synthesis methods (e.g., Niraimathee et al. 2016 [99, 100]). For example, exposure of *Mimosa pudica* root extract to ferrous sulfate (FeSO₄·7H₂O) solution and NaOH resulted in rapid reduction (reaction completed after 20 min at 60 °C) of metal ions and formation of superparamagnetic INPs. The pH of the solution was adjusted to 9 with NaOH which resulted in enhancing the superparamagnetic behavior of the IONs [101].

The semi-green synthesis of INPs by using methanolic extract of *P. tripartita* fruits has been attempted by Kumar et al. (2014), and it was found that Fe_3O_4 nanoparticles were formed after four hours of incubation at 80 °C by using 0.1 M NaOH and 1 mM iron (III) chloride [99]. Prasad et al. (2015) recently reported the addition of $FeCl_3.6H_2O$ and sodium acetate to freshly prepared watermelon rind powder which yielded Fe_3O_4 magnetic nanoparticles [72].

Ehrampoush et al. (2015) reported the formation of IONs from different concentrations of tangerine peel extract, FeCl₃ and FeCl₂·4H₂O in the presence of 25% NH₄OH solution. The synthesized nanoparticles were tested for cadmium removal from aqueous solution. The results have shown that 4 g/L of prepared particles can remove cadmium pollutions with 90% efficiency [67]. *Andean blackberry* leaf extract was used for semi-green synthesis of magnetite nanoparticles by using FeSO₄ as iron precursor at 75–80 °C. [102]. Cao et al. also obtained stable IONP by using the leaf extract of Eucalyptus and cetyltrimethylammonium bromide (CTAB) as a stabilizer in the presence of sodium acetate at 70 °C [103].

A facile and rapid semi-biosynthesis of IONPs was reported by Muthukumar and Manickam (2015) from an *Amaranthus spinosus* leaf extract. The extract was prepared and used for the synthesis of nanoparticles having the rhombohedral phase structure. In this study, IONPs were synthesized by using of aqueous solutions of ferric chloride and pH was adjusted by HCl and NaOH [104]. IONPs were successfully synthesized by Jassal et al. (2016) using extract of *Sapindus mukorossi* (raw reetha) as a natural surfactant. In this work, α -FeOOH and α -Fe₂O₃ were synthesized from ferric nitrate by using potassium hydroxide and sodium hydroxide, respectively. While, the Akaganeite nanoparticles were synthesized from ferric chloride without addition of any alkaline agent [65].

Al-Ruqeishi et al. (2016) demonstrated the rapid synthesis of iron (III) oxide nanorods (IONPs) by using Omani mango tree leave extract and reported that the synthesized nanorods were appropriate for heavy oil cracking process and viscosity treatment. These IONPs were polycrystalline in structure and were gamma phase, maghemite and alpha phase, hematite [105]. The use of aqueous extract of Garlic Vine (*Mansoa alliacea*) leaves in the extracellular semigreen synthesis of iron (III) oxide nanoparticles (β -Fe₂O₃) by using FeSO₄·7H₂O and NaOH was another example recently reported in literature [74].

Other examples of semi-green synthesis of IONPs include the use of *Amaranthus dubius* leave extract using Fe nanoparticles (Fe⁰, FeOOH) through reaction with FeCl₃ solution at continuous stirring for 90 min in the presence of 0.1 N HCl and 0.1 N NaOH [66] and Awwad and Salem (2012) who reported the rapid formation of magnetite nanoparticles when Carob leaf extract was exposed to ferrous chloride (FeCl₂) and sodium hydroxide aqueous solution [106].

Mechanism Behind the Biosynthesis

The basic mechanism of formation INPs, including nucleation and growth of particles, by plant extracts is not obviously discovered. However, studies show that phytochemicals (primary and secondary metabolites) in plant extract play the key role in the biosynthesize of INPs. Actually, these compound especially phenolic compounds (polyphenols, flavonoids, tannic acid, and terpenoids) act as natural antioxidants to impressively reduce iron ions to INPs [66, 69, 77, 91, 100]. FTIR spectroscopy was frequently carried out to investigate the responsible biomolecules for the formation of metal nanoparticles. It is obvious that effective phytochemicals in the formation of INPs are not separate from the prepared particles. These organic compounds make an effective capping on the surface of nanoparticles and improve physicochemical properties of the nanoparticles [35, 88].

FTIR analysis revealed that during the process of nanoparticle generation by the phytochemicals, amines and oxygen bearing functional groups such as phenols, carboxyl, and carbonyl are responsible for the synthesis and capping of INPs [73]. Studies show that plants which have high concentrations of phenolic compounds are the best option for the production of INPs [69, 77, 94, 102]. For instance, spectroscopic studies of the INPs produced by green tea, *Salvia officinalis, Mansoa alliacea, Eucalyptus tereticornis, Melaleuca nesophila, Rosmarinus officinalis, Shirazi thyme*, and pistachio green hulls showed that the main factor in the production of nanoparticles was functional groups of phenolic compounds [74, 76, 95, 96].

Effective Parameters on the Green Synthesis of Iron Nanoparticles

Reaction conditions (i.e., concentration of metal precursors, amount of plant extract in the reaction, reaction temperature, and reaction time) and properties of the plant extract (i.e., pH, antioxidant capacity, extract concentration, and time and temperature of extract preparation) are the key factors that influence physicochemical properties of the synthesized nanoparticles [54, 57, 107, 108]. The effects of each mentioned parameter on the characteristics features of the prepared INPs were examined in various experiments which are discussed in the following sections.

pH of the Plant Extract

The pH of the plant extract is an important factor which influences the stability, size distribution, and type of biosynthesized INPs. It has been shown that plant extracts with different pH (i.e., *H. vulgare* extract with pH 5.8 and *R. acetosa* extract with pH 3.7) produce INPs with different particle size distribution and stability. Plant extract with low pH (*R. acetosa*) produces smaller and more stable nanoparticles as compared to the other extracts. It also has been reported that the presence of organic acids (such as oxalic acid or citric acid) in *R. acetosa* extract increases nanoparticles surface charge and consequently the stability of INPs [70]. In another study, low pH (5.86) of aqueous sorghum extract resulted in the formation of amorphous iron oxyhydroxide nanoparticles which is known to form under acidic condition [63].

Antioxidant Capacity of the Plant

Antioxidant capacity of the plant used for green synthesis is a significant factor as it influences reduction potential of the prepared leaf extract. In general, high antioxidant activity of the plant results in an extract with high reducing capacity which in turn increases the efficiency of synthesized nanoparticle [66, 104]. Antioxidant capacity of a particular plant extracts can be evaluated by using several procedures including Folin-Ciocalteu method, ferric reducing antioxidant power (FRAP) [109, 110], and 2, 2-diphenyl-1-picryl-hydrazyl (DPPH) radical scavenging assay [111]. Several published studies are available which indicate that antioxidant capacity of the prepared plant extract has a linear relationship with the plant dry mass and solvent volume ratio [82, 104]. Various phytochemical compounds with high antioxidant activity such as polyphenols, amaranthine, flavonoids, and amino acids were identified to play an important role in production of INPs [64, 66, 82, 104].

Time and Temperature of the Plant Extract Preparation

Both time and temperature are important factors affecting the formation of nanoparticles as these parameters influence antioxidant capacity of the prepared plant extract. Machado et al. (2013) reported that antioxidant activity of the plant extract increases by increasing the extraction temperature up to 80 °C, however, no significant degradation of the antioxidant compounds at high temperatures [82]. Elsewhere, in another study it was observed that antioxidant capacity of extract increased by elevating the temperature from 40 to 50 °C and at > 70 °C there was a reduction in the antioxidant capacity of the plant extract [66]. It was postulated by the authors that increasing the temperature above 50 °C can degrade antioxidant compounds such as amino acids. The authors also investigated the effect of extraction time from 15 to 60 min and found that the best antioxidant capacity was obtained at 45 min. Due to degradation of antioxidant compounds the extraction time above 45 min was not sustainable. It was also observed that leaf extraction process was not efficient in the heating time less than 45 min [66].

Plant Extract Concentration

Plant extract concentration has a significant effect on the particle size distribution of green synthesized INPs. For

instance, increasing the concentration of tangerine peel extract from 2 to 6% resulted in a significant reduction in INPs size from 200 to 50 nm. However, further increase in concentration of peel extract (up to 10%) induced the agglomeration (strong attachment of nanoparticles) of nanoparticles [67]. Various experiments have been undertaken in recent times which have shown that concentration of antioxidant compounds like amaranthine and phenolic compounds increases by increasing the concentration of plant extract. Variation in the antioxidant capacity of plant extract is known to be the main reason for various effects of plant extract concentration on the characteristic features of green synthesized nanoparticles [66, 67, 104].

Applications of Green Synthesized Iron Nanoparticles

One of the most important applications of INPs is for environmental remediation of pollution (Table 2). The most extensively studied INPs for remediation of water and soil are ZVI NPs due to their favorable catalytic properties [64]. Studies have shown that ZVI NPs can be an effective tool for the treatment of various environmental contaminants such as azo dyes [27], brominated organic compounds [112], antibiotic [113], pesticides [114], nitrate [115], alkaline-earth metals [116], malachite green [77], monochlorobenzene [88], and transition metals such as copper, chromium, and cobalt [117–119]. It has been reported that coating ZVI NPs

with phytochemicals (i.e., phenolic compounds, flavonoids, proteins, and carbohydrates) can enhance the physicochemical characteristics of the fabricated particles [3, 70]. These nanoparticles have so far been tested in few environmental remediation activities. For example, ZVI NPs produced by using the extracts of Shirazi thyme leaf and Pistachio green hulls showed a strong catalytic phosphorus removal activity. The phosphorus removal efficiency was observed to be pH-dependent, and maximum yield was achieved in acidic pH ranges [76].

Fe⁰-iron oxide core–shell nanoparticles that were obtained by using green tea (GT-Fe) and Eucalyptus leaves (EL-Fe) extracts exhibited catalytic nitrate removal activity in swine wastewater. In this comparative study, the reactivity of the green synthesized nanoparticles was measured and was compared with chemically synthesized INPs. Results indicated that green synthesized Fe⁰-iron oxide core–shell nanoparticles have a tremendous potential for environmental remediation [120].

Nanostructures of iron oxides have also gained applications in environmental remediation activities. The IONPs synthesized by *Amaranthus spinosus* leaf extract displayed a strong catalytic activity for decolorization of methylene blue and methyl orange. These particles are more capable for decolorization in contrast to chemically synthesized nanoparticles [104]. Magnetite nanoparticles which were synthesized by using *Andean blackberry* leaf extract have been found to possess a catalytic activity for degradation of organic dyes such as Congo red, methylene blue, and methyl

Table 2 Different INPs from various plants and their potential for organic contaminants removal

Plant	Type of nanoparticles	Contaminants	Initial concentration of contaminant	Removal efficiency (%)	References
Shirazi thyme Pistachio green	Zero-valent iron	Phosphorus	5-300 (mg/L)	97.72–35.79	[76]
Green tea Eucalyptus	Fe ⁰ -iron oxide core–shell	Nitrate	20 (mg/L)	59.7 41.4	[120]
Amaranthus spinosus	IONPs	Methylene blue Methyl orange	100 (mg/L)	69 75	[104]
Andean blackberry	Magnetite	Congo red Methylene blue Methyl orange	10 (mg/L)		[102]
Sapindus mukorossi	α-FeOOH	2-Aminopyridine 3-Aminopyridine 4-Aminopyridine	0.1 mM	75.21 94.27 81.78	[65]
	β-FeOOH	2-Aminopyridine 3-Aminopyridine 4-Aminopyridine	0.1 mM	73.92 92.36 80.12	
	Fe ₂ O ₃	2-Aminopyridine 3-Aminopyridine 4-Aminopyridine	0.1 mM	71.03 88.31 78.99	
Camellia sinensis Red wine	FeNPs	Cr(VI)	50	95 90	[121]
Omani mango	IONPs	Heavy crude oil	_	49	[105]

orange [102]. Catalytic activity is also ascribed to iron oxide hydroxide (FeOOH) nanostructures obtained from *Sapindus mukorossi* (raw reetha). Nanorods of α -FeOOH were found to be highly effective for adsorption of 2, 3, and 4-aminopyridines, and this was attributed to the high surface area of α -FeOOH nanorods [65].

Recently, Mystrioti et al. (2016) tested various plant extracts and juices to find effective sources of phytochemicals for green synthesis of INPs and Cr(VI) reduction. The extract of *Camellia sinensis* (green tea), *Syzygium aromaticum* (clove), and *Mentha spicata* (spearmint) along with *Punica granatum* (pomegranate) juice and red wine were examined as a source of natural reducing agents for iron ions reduction. Pomegranate juice and red wine were identified as the most potent sources of natural polyphenols which are effective in green synthesis of INPs. Prepared nanoparticles were capable to reduce 500 mg Cr(VI) per g of INPs [121].

Green synthesized IONPs have also gained application in heavy crude oil cracking process. Alpha and gamma phase ferric oxide nanorods can increase the efficiency of microwave treatments for reduction in dynamic viscosity of crude oil. Increase in the process efficiency was found to be in direct relation with the concentration of iron oxide nanorods. The crude oil viscosity can be reduced up to 49% by using 0.6 g nanorods per liter crude oil, and this concentration of nanorods was reported to be the saturation point [105].

It is noteworthy that the green synthesized INPs are not safe at all concentrations. The effects of almost all potentially toxic compounds differ in their effects on various organisms and exposure time. Many green synthesized INPs are xenobiotics and could have adverse ecological effects. For example, Markova et al. (2014) investigated the ecotoxicological effects of iron (II, III)–polyphenol complex nanoparticles which were synthesized by green tea extract. In this experiment various concentrations of INPs were exposed to aquatic organisms including *Synechococcus nidulans* (a cyanobacterium), *Pseudokirchneriella subcapitata* (an algae), and *Daphnia magna* (an invertebrate organism). The nanoparticles ecotoxicological impacts were evaluated by ecotoxicological bioassay, and EC₅₀ values were recorded to be in mg per liter concentration ranges [75].

Plant-mediated synthesized INPs are considered as a promising tool for different biomedical applications. The magnetic INPs synthesized by *Albizia adianthifolia* leaf extract utilized for capturing and separation of *Staphylococcus aureus* by a magnetic field. These particles were also effective against human breast carcinoma cell lines. *Albizia adianthifolia*-mediated synthesized INPs are able to induce apoptosis in human breast (both AMJ-13 and MCF-7) cancer cells [122]. In similar study Fe_3O_4 nanoparticles synthesized by *Annona squamosa* leaf extract displayed a cytotoxicity effect against liver carcinoma cell line (HepG2) [123]. Also, INPs with higher oxidation degrease such as Fe_2O_3 which produced by using aqueous extract of *Psoralea corylifolia* have been shown to exhibit significant anticancer activity against renal tumor cells [124].

Recent studies showed that plants-mediated synthesized INPs can be an alternative antibacterial agent to antibiotics. Biologically synthesized INPs using Lawsonia inermis and Gardenia jasminoides leaves extract showed a considerable antimicrobial activity against Escherichia coli, Salmonella enterica, Proteus mirabilis, and Staphylococcus aureus. Results indicated that INPs synthesized by Gardenia jasminoides compared to the INPs synthesized by Lawsonia inermis have a wide zone of inhibition and more potential antimicrobial activity against pathogens [125]. Antibacterial activity is also ascribed to INPs obtained from Sageretia thea (Osbeck.) leaf extract. These nanoparticles show effective antibacterial activity against human pathogenic bacteria's including Escherichia coli, Bacillus subtilis, Staphylococcus epidermidis, Klebsiella pneumoniae, and Pseudomonas aeruginosa [126]. These findings indicate the significant role of phytochemicals in biomedical properties of plant-mediated synthesized nanoparticles.

Importance and Advantages of Plant-Mediated Synthesis

Most of the synthetic methods are potentially hazardous due to the release of noxious chemicals and need to very costly instruments, high-energy, and physical requirements (pressure, temperature). [36]. These issues increase the necessity of developing low costly and more eco-friendly method by means of biological systems like microorganisms (bacteria, yeast, and fungi) and plants [36, 70, 127]. Biosynthesis of metal nanoparticles using plant extract offers significant advantages over other biological systems such as eliminating the cost of cultivation and downstream processing, fast production process and nonhazardous waste [70, 77, 121]. Furthermore, plant-mediated synthesis of INPs is one of the approaches that improve physicochemical and biological properties of nanoparticles [70, 128]. The INPs have high tendency to aggregate and sediment owning to their magnetic attraction properties. Aggregation of INPs is associated with low surface charge and leading to decrease in specific surface area and reactivity of INPs [129]. It has been shown that plant-mediated synthesized nanoparticles are protected with biochemical compound from plant extract which can provide more colloidal stability in aqueous matrixes [54–58, 108, 130].

Feasibility and Future Prospects

In the conventional chemical synthesis of INPs, the most common protocol in the bottom-up method, variety of organic solvents and toxic chemicals such as ammonium hydroxide [59], sodium borohydride (NaBH₄) [131], sodium dodecyl sulfate [132], and hydrazine [9] are used which are not environmental friendly. These techniques utilize flammable reagents and usually are performed at controlled atmospheres, high pressures, and high temperatures [133–135]. Therefore, there is a need for an alternative, economic, and safe method for nanoparticles production [136]. Plant-mediated synthesis of nanoparticles is a green bottom-up technique that does not utilize toxic solvents, high pressures, and high temperatures [137]. However, this method has encountered several important technical challenges including controlling the shape, size and monodispersity of the produced nanoparticles [3]. These problems must be tackled to make plant-mediated synthesis a prosperous and competitive alternative for industrial synthesis of INPs. The type of the used plant is another critical point in regard to the plant-mediated synthesis of metal nanoparticles. In order to choose the best candidates, the effective parameters in the green synthesis such as total protein content and antioxidant capacity of the plant extract need to be determined as these influence the plants capability for nanoparticles production [82]. More research is also required to develop a better understanding on physicochemical properties, activity, and stability of these nanoparticles in order to further improve the practical applications of the plant-mediated INPs.

Conclusion

In this review we represented the ability of different plant species for the green synthesis of INPs. It is certain that plant-mediated synthesis of INPs has significant advantages over the traditional physical and chemical methods. Despite the advantages, plant-mediated synthesis of INPs has significant limitations, especially controlling the shape, uniformity, and monodispersity. These obstacles can be eliminated by optimization studies and controlled reactions. Green synthesis of INPs has so far been carried out at small laboratory scales. Therefore, it is necessary to pay a particular attention for scale-up studies toward industrial production of these nanoparticles in near future.

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