

SYNTHESIS AND CHARACTERIZATION OF IRON OXIDE NANOPARTICLES BY PLANT GREEN METHOD: A REVIEW

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Abstract

The importance of nanomaterials comes from the fact that they possess properties that distinguish them from classical materials, which makes them more efficient in several applications. Therefore, this article discussed one of these nanomaterials, which is nano-iron oxide, and its types (α -Fe₂O₃, γ -Fe₂O₃, and Fe₃O₄) in its particulate form. It focused on the green method and its properties using plant extracts, which are part of a bottom-up methodology for preparing nanomaterials. It also addressed the factors affecting the preparation process, including reaction time, pH, and temperature, through several studies to determine the optimal conditions for preparation. The article then presented the diagnostic techniques used in diagnosing nano iron oxide particles, which are (UV-vis analysis, Fourier transform infrared analysis (FT-IR), X-ray diffraction (XRD), field emission electron scanning microscope (FESEM)) and then reviewed the applications in which the use of nanosized iron oxide was successful, which are toxic organic contamination in the environment, antimicrobial, removal of arsenic from water, antioxidant and anti-radical, and antibacterial

Keywords

Iron oxide NPs, Green synthesis, plant extract, eco-friendly method

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Introduction. Nanoparticles (NPs) have revolutionized various industries and have the potential to address challenges that traditional materials and technologies face. One of the key advantages of NPs is their ability to be engineered and tailored to specific applications [1]. This led researchers to focus on preparing nanomaterials that are distinguished from other materials in terms of their high surface area, stability and controlled effectiveness [2].

The emergence of nanoscience and technology and the unique properties of NPs have opened up exciting possibilities for innovation in medicine, energy, electronics and environmental restoration [3].

In the medical field, NPs have shown promise in targeted drug delivery. By functionalizing the surface of NPs, drugs can be attached or encapsulated within them [4]. Enabling the delivery of the drug to specific and targeted cells or tissues, thus increasing the therapeutic effectiveness of the drug and preventing its side effects [5]. Nanoparticles can also be used in medical imaging for improved contrast and resolution, enabling better visualization of diseased tissues or organs [6].

Nanoparticles also play an important role in enabling renewable energy technologies. In solar cells, for example, NPs can be used to improve light absorption, increase the conversion efficiency of solar energy, and reduce production costs [7]. Similarly, in fuel cells, NPs can serve as catalysts, facilitating faster reactions and improving overall efficiency [8].

The electronics industry has also benefited greatly from nanotechnology [9]. Nanoparticles can be incorporated into electronic devices to enhance their performance, such as improving the conductivity of materials, increasing device stability, and enabling miniaturization [10]. Additionally, NPs can be used in the production of flexible and transparent electronics, unlocking new possibilities for displays, sensors [11], and wearable's devices [12].

Furthermore, NPs offer promising solutions for environmental remediation. It can also be used in various purification processes, such as water filtration and air [13] purification [14], due to their ability to adsorb and remove pollutants [15]. Nanoparticles can also be engineered to degrade harmful chemicals or neutralize toxic substances, making them valuable tools for environmental cleanup [16–18].

However, it is necessary to take into account the potential risks associated with the use of NPs. Their small size and increased reactivity can raise concerns about their potential toxicity and environmental impact. Ongoing research is focused on understanding and mitigating these risks, ensuring the safe and responsible use of NPs in various applications [19–21].

There are various ways to synthesize NPs, which can be broadly classified into two main categories (Fig. 1).

1. The top-down approach: the method includes a methodology for dividing large plates that make up the materials into smaller units, which are then converted into suitable NPs.

2. The bottom-up approach: NPs synthesized through biological system offer many advantages such as no or low toxicity, high productivity, wide applications and controlled shape. Which makes it an innovative method for producing NPs. It can be further divided into different subclasses based on the operation and reaction conditions [22, 23].

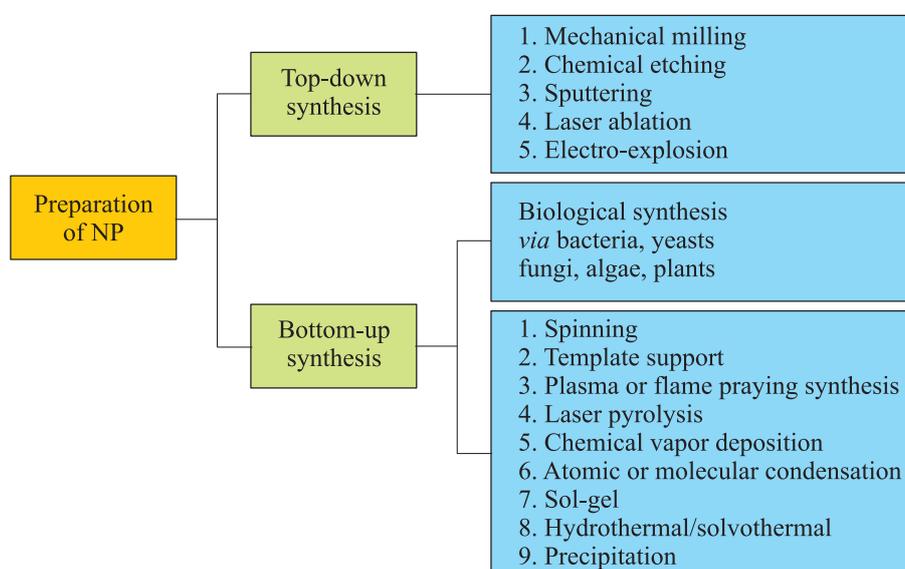


Fig. 1. Nanoparticle synthesis methods

Green approach to iron oxide NPs. The green approach technique has proven to be an efficient method for creating NPs. Nanoparticles prepared by the green method are stable, safe, easy to work with and environmentally friendly. Researchers are particularly interested in the bottom-up synthesis approach using green and biogenic methods due to their practicality and reduced toxicity (Fig. 1). These methods are cost-effective and sustainable as they utilize biological systems such as plant extracts, bacteria, fungi, as well as human cells for NPs synthesis. For example, iron oxide NPs like magnetite (Fe_3O_4), maghemite ($\gamma\text{-Fe}_2\text{O}_3$), and hematite ($\alpha\text{-Fe}_2\text{O}_3$) have been successfully synthesized using green approaches [24, 25]. Over the past decade, the use of iron NPs for environmental restoration has gained traction. Aqueous extracts of some plant residues such as silk hair of corn (*Zea mays L.*) and outer leaves of Chinese cabbage (*Brassica Rapa L. subsp. pekinensis*) are used in the production of nano iron oxide. Extract waste plant as a bio-reducing agent for the synthesis of various metallic [26], semiconductor [27], and antimicrobial activity NPs [28]. Greener approach of iron oxide nanoparticles (INO) has a promising future as it produces materials with non-toxic properties and environmentally friendly behavior. A recent study involved the use of aqueous ferric chloride in the presence of papaya extract to prepare iron oxide nanoparticles. The efficiency of prepared iron oxide NPs as a photocatalyst in degrading Remazol yellow (RR) dye was measured under the influence of several factors such as catalyst dosage, initial dye concentration and pH [29].

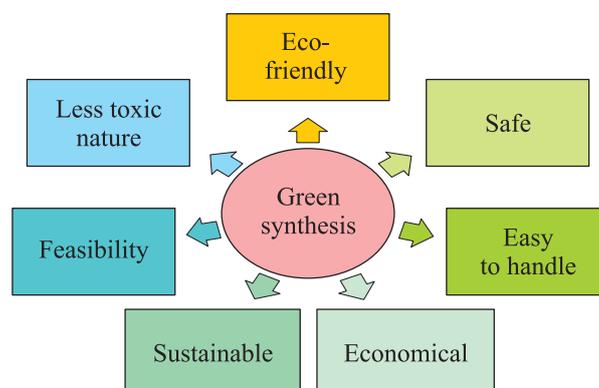


Fig. 2. Green chemistry syntheses properties

In general, plants are available, easy to use, low-cost and non-toxic materials for the manufacture of several types of nanoparticles [30]. This includes extracts of all parts of the plant in the formulation process, such as seeds, flowers, roots, leaves, fruits, peels and petals Fig. 3. Plant sources are rich in various biochemical

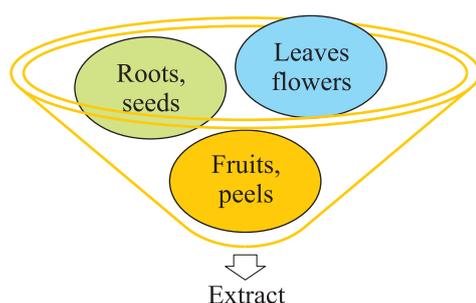


Fig. 3. The sources of plant extracts

such as carbohydrates, amino acids, flavonoids, proteins, saponins, terpenoids and nitrogenous compounds, providing abundant information on active groups. The role of these substances in the green synthesis of NPs is as reducing agents, stabilizers, redox mediators and coating agents. Iron oxide NPs have been successfully synthesized with the help of different plant species through

green biosynthetic pathways, which reflects their bioactivity [29].

Differences between basic chemistry and green chemistry for NPs synthesis. Recently, and for the coming years, green chemistry methods will be more preferable to classical chemistry methods for producing NPs because they are easy to implement, fast, cheap, available, less toxic, and environmentally friendly. The advantages of the green chemical method and the classical chemical method in preparing nanomaterials, including iron oxide NPs, were compared according to Table 1 [31].

General mechanism for NPs synthesis by plant extracts. The biological properties of plants provide biochemical activity to be an efficient cofactor for the synthesis of NPs. The process of obtaining the plant extract is simple to use, as the extract contains many metabolites that act as reducing agents for the synthesis of NPs [32].

**Key differences between basic chemistry and green chemistry
for NPs synthesis**

Classical	Green
High waste accumulation	Prevent waste accumulation
Poor atom economy	High atom economy
Potentially hazardous synthesis	Less hazardous synthesis
Design potentially toxic NPs	Design potentially benign NPs
Use organic solvents and reagents	Use safer solvents and reagents
Apply non-renewable raw materials	Apply renewable raw materials
Use of chemical derivatives	Do not use of chemical derivatives
Use stoichiometric reagents	Use of biocatalysts
Development of non-degradable products	Development of degradable products
High risk of accidents	Lower risk of accidents

The evaluation of the manufacturing process, quality, and stability of the prepared NPs depends on several factors such as pH, temperature, contact time, concentration of metal salts, and phytochemical profile of the plant molecules. The decline of metal ions in plants is faster than that in other living systems because they need to remain in the incubator for a longer time due to the presence of water-soluble phytochemicals. Therefore, the main role of plant extracts is as stabilizing and reducing agents that aid the process of NPs synthesis [32, 33].

Plant extracts contain many biochemical compounds, including proteins and carbohydrates, which act as reducing agents that help in the formation of NPs and the reduction of metal ions. Their importance is gained by their possession of functional amino groups. Also, the functional groups of alkaloids, flavones, and anthracenes, such as $-C-O-C-$, $-C-O-$, $-C=C-$, and $-C=O-$, help in the synthesis of NPs [34].

The chemicals in the plant extract help reduce the metal. By decomposing these chemicals, oxygen is released, which in turn binds to the reduced metal ions. Electrostatic attraction causes the metal oxide ions to bond together, forming NPs. The chemicals then stabilize the NPs at specific levels, preventing them from clumping together as shown in the Fig. 4 [35].

The above can be listed through the following stages.

1. Metals such as iron and titanium tend to form their metal oxides to be in a more stable state through the chemicals of plant extracts.

2. By using the chemicals of plant extracts, the reduction process of metal ions will go through the stages of growth and stability at a safe level.

3. The mechanism of oxygen production is either through the decomposition of the chemicals of the plant extracts or through the atmosphere; it will be linked to a metal ion through stages of growth and stability at a certain level controlled by the chemicals of the plant extracts in order to prevent its aggregation to larger levels, and as a result, the process of producing NPs will be successful [36, 37].

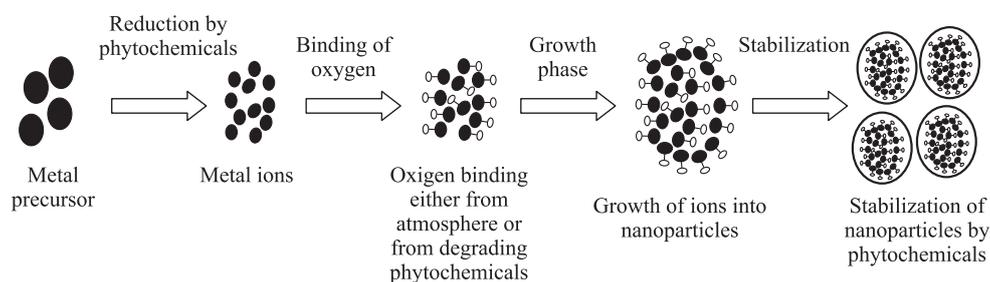


Fig. 4. Metal and metal oxide NPs formation mechanism by phytochemicals

Factors impact on the green synthesis of iron oxide NPs. Preparation of various NPs using environmentally friendly methods is influenced by factors such as pH, temperature, reaction time, and reactant concentration. These parameters play a crucial role in determining the nanoparticle morphology and optimizing the synthesis process. Additionally, they have a significant impact on the environmental factors involved in the synthesis of metallic NPs.

Temperature impact. Temperature plays an influential role in controlling the properties (size, composition, and shape) of iron oxide NPs across various research studies around the world. Researchers can adjust the temperature and thus set the shape and size of nanoparticles in different patterns, such as spheres, octahedral platelets, rods, triangles, and other shapes. The increase in temperature increases the reaction response rate and speed, which accelerates the formation of these nucleation centers on the one hand. On the other hand, during the green synthesis of NPs, the reaction time at a certain temperature appears as an important factor affecting the shape, size, and yield of the synthesized NPs, which has been mentioned in many studies as in Table 2.

A specific study involved the green preparation of nano iron oxides using plant extracts of *Phoenix dactylifera* (PDL) over a wide temperature range. The results showed that temperature plays an important role in the synthesis process. Therefore, low calcination temperatures over a short period prevent the

Table 2

Optimal conditions for preparation of iron oxide NPs

Name of plant (common)	Part used	pH of reaction	Temperature dry or annealing, °C	Time of reaction, min	Reference
<i>Source of iron oxide NPs FeCl₃·6H₂O</i>					
<i>Ficus Carica</i>		11.0	80	60	[29]
<i>Daphne mezereum</i>	Leaves	7-8	600	60	[42]
<i>Phoenix dactylifera</i>			400	60	[43]
<i>Mentha Piperita</i>			25	30	[44]
<i>Source of iron oxide FeSO₄·7H₂O</i>					
<i>Laurus nobilis</i> L.	Leaves	7-8	500	150	[45]
<i>Source of iron oxide FeSO₄·7H₂O + FeCl₃</i>					
Leaves of (black tea, oak tree, green tea, pomegranate, and eucalyptus)	Leaves	6.0	80	20-60	[46]
<i>Source of iron oxide FeCl₃</i>					
<i>Sageretia thea</i> (Osbeck.)	Leaves	5.7	500	60	[47]
<i>Spinacia oleracea</i> (spinach), <i>Musa acuminata</i> (banana)	Spinach leaf, Banana peel	5.0	-	30	[48]
<i>Carica papaya</i>	Leaves	7-8	400	50	[49]
<i>Eucalyptus</i>		3	50	60	[50]

End of the Table 2

Name of plant (common)	Part used	pH of reaction	Temperature dry or annealing, °C	Time of reaction, min	Reference
<i>Neem (Azardica indica)</i>	Leaves	8-12	60 / 250	360 / 120	[51]
	Source of iron oxide $\text{FeCl}_3 \cdot 6\text{H}_2\text{O} + \text{FeCl}_2 \cdot 4\text{H}_2\text{O}$				
<i>Prosopis Africana</i>	Leaves	9.0	70	35	[52]
	Peel	-	25	30	[53]
<i>F. Carica Fruit</i>	Source of iron oxide Iron (III) chloride hexahydrate				
	Leaves	-	60	120	[54]

decomposition of the active ingredients. This leads to the formation of NPs with smaller sizes, fewer polycrystalline structures, and better functional properties. The opposite is true [38]. In addition to the above, it should be mentioned that heat shock proteins are produced by microorganisms under high-temperature conditions, which helps in the successful synthesis of metal and metal oxide NPs using the green method [39].

pH impact. The role of pH in shaping NPs is crucial. pH controls the creation of the nucleation process. Increasing the pH level automatically increases the number of nucleation centers, which is crucial for enhancing the formation of metal NPs. It is well-known that pH plays an important role in determining the size and structural morphology of NPs. The pH of the medium is a key determinant in the formation of NPs, which is mentioned by several studies as in Table 2.

In general, the basic pH (> 7) helps in preparing the functional groups to reach and bind to them in the reaction medium, which leads to the formation of the nucleus and the continuation of the growth process until the construction of NPs. This is confirmed according to what was previously mentioned that the alkaline medium in conjunction with protein molecules has an important role in the synthesis of various NPs [40]. On the other hand, a study confirmed the association of an increased number of nanoparticles manufactured in a green way by extract of *Cinnamomum zeylanicum* bark and high pH values. Bio-synthesis methods of mineral/metallic materials and their oxides with organized micro/nanostructures are important in both theoretical concepts and practical applications due to their low toxicity, low pollutants as by-products, and low energy consumption. Biochemicals extracted from living systems have a great ability to convert metal ions into biominerals and bioreducibly produce nanocrystalline materials of various shapes and sizes, which reflects the improvement of the safety and sustainability of nanoparticle production [41].

Reaction time impact. The structural morphology of NPs is primarily influenced by reaction time, temperature, and pH. In the synthesis of magnetic NPs, the significance of reaction time cannot be overstated, which mentioned by several studies as in the Table 2.

Precursor concentration impact. Molar ratios of reactants are important parameters that affect the size of NPs during the chemical synthesis process. Their effect is directly represented in the products of the preparation process. This was confirmed by a study that included the possibility of controlling the shape of the nanocrystals synthesized by the green method by controlling the change in the concentration of the reactant. Another study also confirmed that

increasing the production of bio-organic materials from plant extracts increases the size of the resulting NPs. It is also possible that precursors with a higher molar ratio also have an effect on the shape of NPs [42].

Various techniques have been utilized to identify the types of iron oxide NPs synthesized due to the aforementioned factors. These techniques include FESEM, EDX, XRD, FT-IR, and UV-vis spectroscopy. Several studies have also evaluated the efficacy of these NPs, with the findings presented in a Table 3.

Advantages and drawbacks of iron oxide green synthesis. Green synthesis has attracted great attention in the field of nanomaterials manufacturing. It is more useful than traditional synthesis methods because it has several distinctive characteristics, being low-cost, environmentally friendly, and non-toxic, in addition to requiring moderate reaction conditions, such as low temperatures and pressures. As a result, energy consumption is reduced and overall safety and process stability are enhanced. However, on the other hand, green synthesis may not be suitable and available for all types of reactions or desired products, because some complex reactions or molecules may require harsher conditions or specific reagents that are incompatible with the principles of green synthesis. Therefore, green methodological preparation methods require prior knowledge and understanding of their principles and mechanisms so that they do not become an obstacle for researchers when applying them in complex reactions and unfamiliar products, as the green synthesis of NPs has many advantages and disadvantages that are listed in the Table 4 [55].

Applications of NPs. Nanoparticles have superior properties that qualify them for application in many areas of life in Table 2. The synthesis of NPs using plant extracts has many applications in catalysis, drug delivery, biotechnology medicine, and water treatment. Nanoparticles have a precise sensitivity as they can be detected by imaging tools and devices, which makes them suitable for imaging, therapy, and drug delivery. Nanoparticles have different dimensions, which makes them have different biomedical uses. In other areas, such as environmental applications and water treatment, metal nanoparticles and their oxides play an important role due to their unique properties and high surface area-to-volume ratio. This makes them effective in removing organic and inorganic contaminants from water sources. However, challenges remain, such as concerns about toxicity, agglomeration, and cost-effectiveness, that need to be addressed in order to expand their use [38, 56–58].

Conclusion. Optimal conditions for the synthesis of Fe–O nanoparticles are considered in detail in order to obtain the desired properties and increase the yield of the product. According to literature, we conclude from the above article the success of using various plant extracts in implementing the green

Table 3

Characterization and applications of iron oxide NPs

λ_{max} nm, by UV-vis data	Size by XRD data, nm	2 θ by XRD data, °	Fe-O by FT-IR data, cm^{-1}	Shape by FESEM analysis	Activity	Reference
291, 206	43-57	14, 27, 49	400-570, 620-660, 470, 540	$\gamma\text{-Fe}_2\text{O}_3$ / $\alpha\text{-Fe}_2\text{O}_3$ Agglomerated and are multiform	Antioxidant	[29]
465	100	20-45	516.9	Fe_2O_3 / Fe_3O_4 Spherical	Toxic organic contamination in the environment	[42]
$\alpha\text{-Fe}_2\text{O}_3$ NPs						
285	60	33.2, 35.6	600, 450	Hexagonal shape	Anti-microbial	[45]
230-240	35-100	38	598, 484	Fe_3O_4 , $\gamma\text{-Fe}_2\text{O}_3$, $\alpha\text{-Fe}_2\text{O}_3$ Spherical	Removal of arsenic from water	[46]
225, 275	2.3-22.16	32-61	573, 638	$\alpha\text{-Fe}_2\text{O}_3$ / Fe_3O_4 Spherical	Antioxidant and anti-radical	[43]
514	~ 29	25-40	~ 500, 772	$\gamma\text{-Fe}_2\text{O}_3$ Tetragonal crystalline shape	Antibacterial	[47]

End of the Table 3

λ_{max} nm, by UV-vis data	Size by XRD data, nm	2 θ by XRD data, °	Fe-O by FT-IR data, cm ⁻¹	Shape by FESEM analysis	Activity	Reference
Fe NPs						
240–430, 240, 270, 395	20–50, 10–70	18.15–47.55	540, 460	–	Antibacterial potential, <i>in vivo</i> toxicity evaluation	[48]
$\text{Fe}_3\text{O}_4 / \text{Fe}_2\text{O}_3$						
300, 450	277, 29, 45	–	584, 402, 687, 501	Spherical and cubic	Antimicrobial	[51]
$\alpha\text{-Fe}_2\text{O}_3$						
430	8.8	27.71, 35.86, 56.6	587.85, 566.86, 543.88	–	<i>In vivo</i> toxicity evaluation	[44]
Fe_2O_3						
390	43	–	–	Spherical	Antibacterial	[49]
360	–	21.9–62.4	1098, 795, 506, 463		Anti-microbial	[54]
$\text{Fe}_3\text{O}_4\text{-NPs}$						
400, 633	30–100	–	404	Spherical	Antibacterial	[52]
610	55	35–55	571	Crystal phase	Decolorization of dye	[50]
417–440	30–40	21.33–43.35	–	Semi-spherical	Antibacterial	[53]

Advantages and disadvantages for green synthesis of NPs

Advantages	Disadvantages
Low cost	Nanotechnology has increased the potential for risks to human health
Facile implementation	Lack of studies on the biological and toxicological deposits of nanoparticles in the environment
Eco-friendly approach, toxic solvents, and chemicals are excluded in this method	Nanoparticles are dangerous to the human body, as due to their small size, they can easily enter the body and cause many health problems
No contamination compared to classical methods (chemical and physical synthesis)	Industrially, compared to chemically generated NPs, green synthesis has limited use
Ability to control the synthesis of NPs of specific shape and size	
Recycling and reusing waste materials	
It requires moderate conditions of pressure and temperature	
Possibility of using available and renewable raw materials	
Its effectiveness covers a wide area in the production of NPs	

method for preparing iron oxide NPs of various types (γ -Fe₂O₃, α -Fe₂O₃, Fe₃O₄) with a spherical shape and a size of less than 100 nm and a maximum absorption peak ranging within the region 230–500 nm and at diffraction angles 25°, 35°, and 45°, and an effective group for iron oxide appears at a wavenumber ranging from 400–660 cm⁻¹, based on the results of techniques (FT-IR, XRD, UV-Vis, FESEM). It has also been proven that the prepared iron oxide has high efficiency in several applications: toxic organic contamination in the environment, anti-microbial, removal of arsenic from water, antioxidant, antiradical, and antibacterial.

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