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Plant-Mediated Synthesis of Iron Oxide Nanoparticles Using Ferula persica

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ABSTRACT

Iron nanoparticles are among the most important metallic nanoparticles composed of iron. Due to their extremely small size, low toxicity, and excellent magnetic properties, iron nanoparticles have demonstrated outstanding applications compared to similar nanoparticles. One of the best alternative approaches for the synthesis of iron nanoparticles is the use of green synthesis methods. Therefore, the approach of this study was the green synthesis of iron nanoparticles. In this research, leaf extracts of plant *Ferula persica*, were used for the green synthesis of iron nanoparticles. XRD, FT-IR, and FESEM techniques were employed to investigate and characterize the synthesized iron nanoparticles. Based on the UV–Vis spectrum, for *Ferula persica* the concentration was 50 mM. The optimal extract concentration for *Ferula persica* plants was 4%. This can be attributed to higher absorption and better peak broadening at optimal concentrations. The synthesized nanoparticles from the *Ferula persica* plant sample had an average size of 55 nm.

INTRODUCTION

Iron nanoparticles are particles with sizes ranging from 1 to 100 nm that are composed of iron. Iron-based nanomaterials are low-cost and widely available [1]. Zero-valent iron, bimetallic/trimetallic iron-based compounds, and iron oxide/hydroxide/oxyhydroxide-based compounds constitute the main categories of iron-based nanomaterials. Among various iron-based nanomaterials, zero-valent iron has attracted particular attention due to its unique properties [2]. Zero-valent iron nanoparticles are highly reactive and iron is the fourth most abundant metal in the Earth's crust.

Iron oxide nanoparticles, owing to their very small size, suitable magnetic properties, and high biocompatibility, are considered highly promising candidates for applications in various fields, including data storage, imaging, medicine, photocatalysis, molecular diagnostics, and targeted drug delivery [3].

Among the different structures of iron oxide, the alpha phase of this material has received considerable attention in environmental applications [4]. Meanwhile, the gamma phase is a highly suitable option for use in data storage and drug delivery. In recent years, iron nanoparticles, due to their excellent magnetic properties compared to other similar iron oxide-based nanoparticles, have demonstrated outstanding applications successfully. The toxicity of iron nanoparticles is lower than that observed for many other types of nanoparticles [5].

To date, numerous chemical methods have been reported for the synthesis of iron nanoparticles; however, due to the significant drawbacks associated with chemical approaches, there is a fundamental need for nanoparticle production via green chemistry methods, which are environmentally friendly and sustainable [6]. According to this approach, extracts of medicinal plants containing bioactive compounds can simultaneously act as reducing agents for iron ions and as chelating (complexing) agents, leading to the synthesis of zero-valent iron nanoparticles [7].

Plants naturally produce a wide variety of compounds with different chemical natures that are utilized for their growth and development [8]. Primary metabolites provide the essential resources required for processes such as photosynthesis, transport, and respiration[9]. Compounds derived from primary metabolites that are not directly involved in growth and development are considered secondary metabolites.

In other words, secondary metabolites are small organic molecules that originate from primary metabolites in plants, and their chemical nature and composition vary among species [10]. Secondary metabolites generally possess complex structures, which differ from the relatively simpler structures of primary metabolites that are essential for cell survival. The three main classes of secondary metabolites include phenolic compounds (consisting of simple sugars and benzene rings), terpenes

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and steroids, and nitrogen-containing compounds [11]. Phenolic compounds are synthesized via the phenylpropanoid pathway in plants, exhibit diverse structures, and are responsible for the coloration of flowers, fruits, and vegetables [12]. Alkaloids and flavonoids are large groups of secondary metabolites with highly diverse biological functions [13].

In 2022, Salman Haider et al., conducted a study in which they synthesized iron oxide nanoparticles using tea waste. The synthesized material was then applied as a micronutrient nanofertilizer as a substitute for conventional iron fertilizers, and its effects on the growth and development of mulberry (*Morus alba* L.) plants were evaluated. The synthesized iron nanoparticles were applied at two different doses (10 and 50 mg per kg of soil) through soil application and foliar spraying. The application of iron oxide nanoparticles at 10 mg per kg of soil significantly improved plant morphological traits, including increased root length and a reduced time interval for the emergence of the first leaf [14].

In 2017, Kumar et al., aimed to synthesize iron oxide nanoparticles using a cost-effective and environmentally friendly method. They prepared iron oxide nanoparticles via a green chemistry approach and evaluated the biological activities of the synthesized nanoparticles [15]. In this method, *Vigna mungo* plant extract and iron sulfate at different concentrations were used for the synthesis of iron oxide nanoparticles. The analysis results indicated that the average size of the synthesized iron nanoparticles ranged between 10 and 20 nm. Additionally, these nanoparticles exhibited rod-like, crystalline, and highly stable structures. The antibacterial activity of the synthesized iron oxide nanoparticles was also analyzed.

In 2022, Ndaba et al., used African wormwood (*Artemisia afra*) leaf extract for the green synthesis of iron oxide nanoparticles and investigated their effects on the seed germination of spinach and carrot. X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD) measurements were employed to characterize the synthesized iron nanoparticles. The results of these analyses indicated that the iron oxide nanoparticles had sizes ranging from 15 to 20 nm [16].

Ferula persica is a well-known species of the *Ferula* genus that is widely used in the treatment of diseases such as diabetes, rheumatism, and back pain [17]. As shown in Figure 1, this plant is characterized by robust, hollow, and somewhat succulent stems reaching up to one meter in height, along with yellow flowers, and it grows in regions of Iran, Turkey, and Afghanistan [17].

In Iran, this plant is known by its Persian name "Koma" and is considered one of the endemic (native) plants of the country. Due to its limited distribution and various threats to Iran's ecosystems, it is currently regarded as an endangered species. *Ferula persica* is a perennial and monocarpic plant, meaning it flowers and fruits only once before dying.

Fig. 1. An image of the flower, leaf, and whole plant of Ferula persica.

A comprehensive review of the literature indicates that there are no reported studies on the synthesis of iron nanoparticles using *Ferula persica* extract. Therefore, in this study, considering the properties and characteristics of these plant extracts, iron nanoparticles were synthesized using these compounds. Subsequently, the synthesized nanoparticles were compared in terms of their morphology and size. Accordingly, the main objective of this research is the synthesis of iron oxide nanoparticles using a cost-effective and environmentally friendly method.

MATERIALS AND METHODS

Extraction of *Ferula persica* extract

Leaves of *Ferula persica* was collected from region of Kurdistan Province. *Ferula persica* leaves were gathered from the highlands of Bijar toward Takab. The plant materials were then dried and powdered. A total of 10 g of powdered leaves of *Ferula persica* were accurately weighed. Separately, 100 mL of distilled water was added to each sample. Each mixture was stirred for 24 hours. The obtained extracts were then filtered using Whatman No. 42 filter paper

Optimization of Parameters Affecting the Synthesis of Iron Oxide Nanoparticles

To achieve the optimal synthesis method and maximize the yield of synthesized iron nanoparticles, the most influential parameters affecting the synthesis process were evaluated and optimized. In general, these parameters included the concentration of $FeCl_3.6H_2O$ salt and the concentration of the plant extract.

Optimization of $FeCl_3.6H_2O$ Salt Concentration

For the optimization of $FeCl_3.6H_2O$ concentration, four different concentrations of the iron salt were prepared: 5 mM, 10 mM, 20 mM, and 50 mM. Each of these solutions was then separately added to a fixed concentration of the extract from each plant. Subsequently, the UV–Vis spectra of each solution were recorded.

Optimization of Extract Concentration of *Ferula persica*

To optimize the extract concentration, five different concentrations of each plant extract were prepared separately within a concentration range of 0.5% to 4%. Each extract was then mixed with a $FeCl_3.6H_2O$ solution at a fixed concentration of 10 mM, and the UV–Vis spectra of each resulting solution were recorded.

Synthesis of Iron Oxide Nanoparticles Using Extracts of *Ferula persica*

For the synthesis of iron oxide nanoparticles, 2 mL of *Ferula persica* plant extracts were separately mixed with 4 mL of a 50 mM $FeCl_3.6H_2O$ solution. Upon the addition of the $FeCl_3.6H_2O$ solution, a color change was immediately observed in the reaction mixture; specifically, the solution changed from yellow to dark brown, indicating the formation of iron oxide nanoparticles.

To collect the synthesized iron oxide nanoparticles, the solution was centrifuged for 15 minutes at 3000 rpm. The precipitated iron oxide nanoparticles were then separated and dried in an oven at 30 °C. The resulting powder was further subjected to calcination in a furnace at 450 °C for 4 hours to complete the growth and crystallization process of the synthesized nanoparticles. Finally, the dried sample was collected and stored in appropriate containers.

RESULTS AND DISCUSSION

Investigation and Optimization of Parameters Affecting the Synthesis of Iron Oxide Nanoparticles

Effect of Iron Salt ($FeCl_3.6H_2O$) Concentration

For the optimization of $FeCl_3.6H_2O$ concentration, four different concentrations of the iron salt (5 mM, 10 mM, 20 mM, and 50 mM) were prepared. Then, a fixed volume of plant extract and a fixed volume of the iron salt solution, equivalent to 2 mL and 4 mL respectively, were separately added to each of these solutions.

Based on the UV–Vis spectra corresponding to *Ferula persica*, it can be stated that the optimal iron salt concentration for samples was 50 mM.

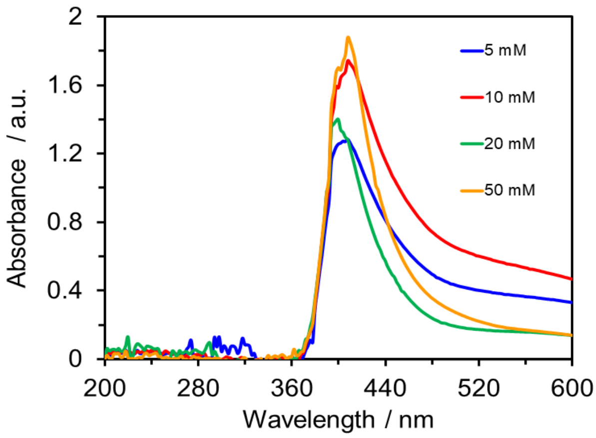

Fig. 2. UV–Vis spectra of *Ferula persica* at iron salt concentrations of 5 mM, 10 mM, 20 mM, and 50 mM.

Effect and Evaluation of Plant Extract Concentration

For the optimization of plant extract concentration, five different concentrations of each extract were initially prepared separately within the range of 0.5% to 4%. Then, a fixed volume (2 mL) of plant extract and a constant volume (4 mL) of the iron salt solution were separately added to each of these solutions.

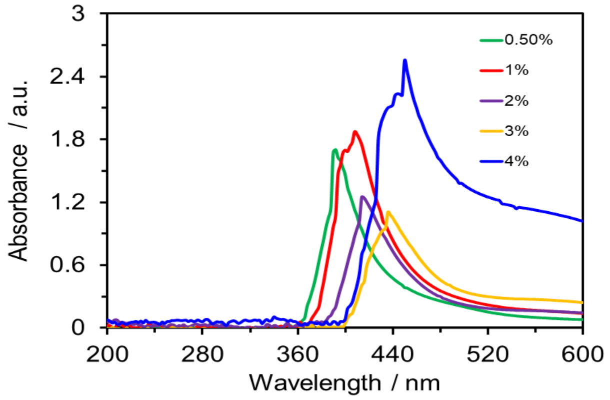

Fig. 3. UV–Vis spectrum of *Ferula persica* extract at concentrations ranging from 0.5% to 4%.

XRD Pattern Analysis

To confirm the success of the synthesis and to determine the crystalline structure of the synthesized iron compounds, XRD analysis was performed over a 2θ range of 5°–80°. The XRD pattern obtained for the iron synthesized using *Ferula persica* extract is shown in Figure 4.

In general, the presence of characteristic peaks corresponding to the components involved in the synthesis indicates the successful formation of iron particles. Based on analysis using X'Pert HighScore Plus software and according to the JCPDS reference card No. 00-033-0664, the main observed peaks at 24.13°, 33.15°, 35.61°, 49.48°, 54.09°, 62.45°, and 63.99° correspond to the characteristic diffraction peaks of iron oxide (Fe_2O_3) with a rhombohedral crystal system.

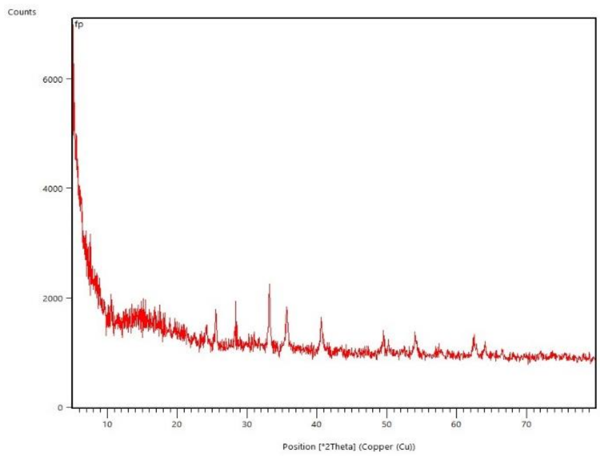

Fig. 4. XRD pattern of iron nanoparticles synthesized using *Ferula persica* extract.

Furthermore, the average particle size of the synthesized nanoparticles was calculated to be approximately 54 nm using the Debye–Scherrer equation.

FT-IR Spectral Analysis

To investigate the chemical structure of the synthesized iron nanoparticles, FT-IR spectra were recorded in the wavenumber range of 400–4000 cm^{-1} using potassium bromide (KBr) pellets. Figures 5(a) and (b) show the FT-IR spectra of the iron nanoparticles synthesized using *Ferula persica* extract and the plant extract itself, respectively.

In Figure (a), a strong characteristic absorption peak at 514 cm^{-1} corresponds to the Fe–O stretching vibration. Additionally, the absorption peak observed at 1398 cm^{-1} is attributed to asymmetric C–O stretching vibrations of the carboxyl group. In both spectra (a) and (b), the appearance of absorption peaks at 1257 cm^{-1} and 1266 cm^{-1} is associated with C–N stretching vibrations. The peaks at 1627 cm^{-1} and 1614 cm^{-1} indicate C=O stretching vibrations of carboxyl groups.

Moreover, the absorption bands appearing at 2926 cm^{-1} and 2927 cm^{-1} are related to C–H stretching vibrations. The peaks at 3431 cm^{-1} and 3421 cm^{-1} correspond to O–H stretching vibrations of hydroxyl functional groups [18].

The green synthesis of iron oxide nanoparticles using *Ferula persica* extract is likely mediated by various bioactive secondary metabolites present in the plant extract, including phenolic compounds, flavonoids, terpenoids, and other antioxidant molecules. These phytochemicals may act as both reducing and stabilizing agents during nanoparticle formation. Hydroxyl and carbonyl functional groups identified in the FT-IR spectra suggest their involvement in the reduction of Fe^{3+} ions into iron oxide nanoparticles and in preventing particle agglomeration through surface capping and stabilization. In particular, phenolic compounds and flavonoids are known to donate electrons and facilitate the bioreduction process, leading to the formation of stable nanoparticles with nanoscale dimensions.

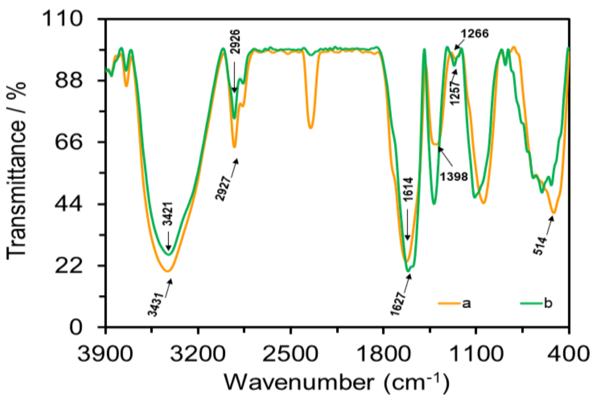

Fig. 5. (a) FT-IR spectrum of iron nanoparticles synthesized using *Ferula persica* extract, and (b) FT-IR spectrum of *Ferula persica* extract.

FESEM Image Analysis

FESEM analysis was employed to examine and confirm the formation of iron nanoparticles. As shown in Figure 6, FESEM images of the synthesized iron nanoparticles clearly indicate that the particle size is below 100 nm, confirming the formation of nanoparticles at the nanoscale.

Additionally, the images demonstrate a relatively uniform distribution of the nanoparticles. The iron nanoparticles synthesized using *Ferula persica* extract exhibit a compact structure with a cubic morphology. Fadian et al. investigated the synthesis of magnetic iron oxide nanoparticles using an alcoholic extract of the marine alga Sargassum ilicifolium [19]. The results obtained from UV–Vis spectroscopy, XRD, and TEM analyses confirmed the synthesis of magnetic iron oxide nanoparticles using the algal extract. The formation of iron oxide nanoparticles was accompanied by a color change from green to brownish-green.

The maximum absorption of the nanoparticles was observed at around 821 nm, indicating the reduction of iron ions and the formation of iron nanoparticles. According to the results of this study, the nanoparticles exhibited a spherical morphology with a face-centered cubic crystalline structure and an average size of 22.09 nm. Furthermore, these nanoparticles demonstrated remarkable superparamagnetic and soft magnetic properties, which are unique among iron-based nanoparticles.

Firouzi et al. utilized leaf extracts from Eucalyptus globulus for the green synthesis of iron nanoparticles from iron(III) chloride, characterized by UV-Vis, FTIR, FESEM/EDS techniques with particle sizes around 50 nm [20]. The results indicated that the most effective iron nanoparticles were produced using acacia plant extract. Accordingly, they reported that iron nanoparticles can be synthesized using an aqueous extract of acacia combined with suitable aqueous solutions of iron salts such as iron oxide and iron chloride.

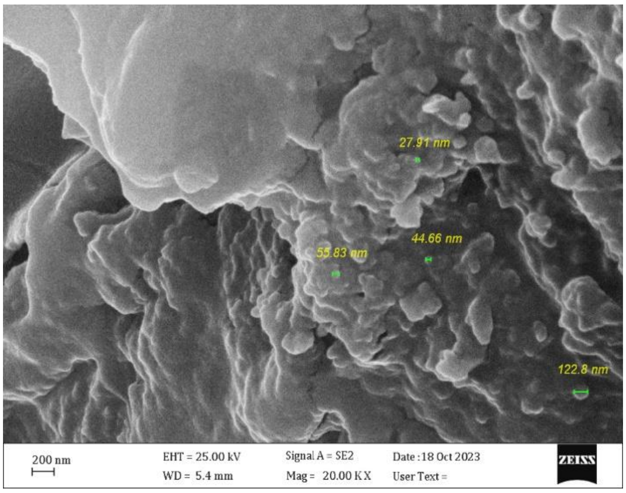

Fig. 6. FESEM images of iron nanoparticles synthesized using *Ferula persica* extract.

CONCLUSION

In this study, an environmentally friendly and cost-effective green synthesis approach was successfully employed for the production of iron oxide nanoparticles using plant extracts of *Ferula persica*. The findings demonstrated that plant derived biomolecules, including phenolic compounds, flavonoids, and other secondary metabolites, play a crucial role as reducing and stabilizing agents in the nanoparticle synthesis process.

Optimization of key synthesis parameters, including the concentration of $FeCl_3 \cdot 6H_2O$ and plant extract, revealed that a salt concentration of 50 mM and an optimal extract concentration significantly enhanced nanoparticle formation, as confirmed by UV–Vis spectroscopy. Structural and morphological characterization using XRD, FT-IR, and FESEM analyses verified the successful synthesis of iron oxide nanoparticles with a rhombohedral crystalline structure, an average particle size in the nanoscale range (~50–55 nm), and a relatively uniform distribution with compact, cubic-like morphology. FT-IR results further confirmed the involvement of functional groups such as hydroxyl, carbonyl, and amine groups in the reduction and stabilization processes.

Comparative analysis with previous studies indicates that the synthesized nanoparticles exhibit appropriate particle size, stability, and potential magnetic behavior, while benefiting from reduced toxicity and sustainable production methods. The absence of prior reports on the use of *Ferula persica* extract for iron nanoparticle synthesis highlights the novelty of this work.

Overall, this study demonstrates that *Ferula persica* extract can serve as an efficient bioresource for the green synthesis of iron oxide nanoparticles, offering promising potential for applications in environmental, biomedical, and nanotechnological fields. Future studies can focus on exploring their functional performance in specific applications such as catalysis, drug delivery, and environmental remediation.

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