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Biosynthesis and Characterization of Fe Nanoparticles with Quinoa Leaf Extracts: Potential for Environmental Remediation

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The biosynthesis of iron nanoparticles with quinoa leaf extracts represents a breakthrough in green nanotechnology, offering an environmentally friendly method that improves biocompatibility. The objective of the research was the biosynthesis of Fe nanoparticles (NpFe) with quinoa leaf extracts and their respective characterization, through the recognition of functional groups, composition, crystalline structure and morphology of the nanoparticles, using FTIR, X-ray fluorescence, X-ray diffraction and TEM analysis, respectively. Nanoparticles with diameters ranging from 1 to 100 nm and composed of 74.4 % oxygen and 21.1 % iron were obtained, presenting an amorphous structure influenced by the plant matrix. Their coating with OH^- groups derived from phenolic compounds increases their potential in a wide range of applications, from drug delivery and cancer therapies to environmental remediation and the development of advanced sensors. Furthermore, their potential in agriculture to enhance nutrient absorption and as remediation agents underscores their versatility. This sustainable approach not only reduces the use of harmful chemicals, but their characteristics also give nanoparticles advantages and new possibilities in various scientific and technological fields.

1. Introduction

In the face of heavy metal contamination problems, nanoparticles have emerged as a highly effective solution for the remediation of these contaminants in various environmental matrices such as water and soil, due to their unique physicochemical properties, such as high surface area, reactivity, adsorption capacity, and selectivity, which make them especially suitable for remediation; among them, iron nanoparticles have precipitation, reduction, and oxidation mechanisms (Yadav et al., 2024) (Manwani et al., 2024). Nanoparticles can be used in a variety of forms (Maryam and Gul, 2023) and have high efficiency attributed to their small size and large surface area, which increase contact with contaminants (Damiri et al., 2022). Biogenic nanoparticles synthesized from plant extracts have shown great promise in removing heavy metals (Real and Benites, 2021) (Benites-Alfaro et al., 2023) (Gómez et al., 2023).
The metal removal mechanisms of nanoparticles are primarily based on their adsorbent properties, large surface area, and functional groups (Lee et al., 2019). They sometimes act by reducing metals to their less toxic form or catalyzing their degradation, as is the case with iron nanoparticles. These nanoparticles are characterized by their easy separation using an external magnetic field, which facilitates their recovery and reuse (Kumari et al., 2020).

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Nanoparticles can be used in the remediation of contaminated soils, where they have demonstrated high efficiency in immobilizing heavy metals (Kumari et al., 2020). While nanoparticles offer significant advantages, their use must be cautious, considering potential environmental risks and toxicity, and their safe and sustainable use must be assumed (Shwetha and Ramanna, 2023).
Within this range of applications, to achieve this safely and overcome some disadvantages, the research objective focused primarily on the synthesis of nanoparticles with environmental characteristics from a plant extract of quinoa leaves. This would allow obtaining nanoparticles that can act by exploiting the synergistic effects of their structure.

2. Methodology

To obtain iron nanoparticles by reducing ferric chloride with phenolic extract of quinoa leaves, the following steps were followed:

2.1 Preparation of quinoa leaf extract

- 200 g of fresh quinoa leaves were collected and washed.
- The quinoa leaves were dried at 50 °C for 24 hours.
- The leaves (5 g) were placed in a 1-liter beaker. To prepare the extract, a 1:10 % w/v ethanol solution was added to the quinoa leaves and left to stand for 24 hours.
- The extract was then filtered to separate the solution.
- This extract was subsequently analysed.

2.2 Biosynthesis of iron nanoparticles

The procedure followed was:

- Initially, 100 mL of a 0.1 M $FeCl_3$ solution was prepared in an Erlenmeyer flask, yielding a yellow solution.
- Subsequently, 25 mL of the 0.1 M FeCl3 solution was taken and 25 mL of quinoa leaf extract was added drop by drop (1:1 ratio), observing the colour change from yellow to black. The pH was then measured, recording a value of 3.12. NaOH 1 M was added to adjust the pH to 4.12, as scientific literature states that a pH in the range of 4 to 5 is optimal for obtaining nanoparticles (Kumar et al., 2015 and Mystrioti et al., 2016). At that moment, the solution turned a deep black colour, and the presence of a precipitate was observed (see Figure 1).
- The solution was then centrifuged at 10,000 rpm in 50 mL Falcon tubes for 10 minutes.
- The precipitate was washed three times with a 50 % v/v acetone-alcohol solution, then dried for 24 hours at 50 °C (see Figure 2). The precipitate was weighed giving 0.5 g.
- The precipitate obtained was iron nanoparticles, which were then characterized accordingly and stored in an amber glass bottle at room temperature.

Figure 1: Black precipitate, corresponding to iron nanoparticles in quinoa extract.

Figure 2: Iron nanoparticles

2.3 Mechanism of formation of iron nanoparticles:

When mixing the FeCl3 solution with the polyphenols contained in the plant extract of quinoa leaves, the mechanism of chemical balance of Eq. (1) is presented.

$$2Fe^{+3} + 3R\text{-}C_6H_3(OH)_2 \rightarrow Fe^{\circ} + FeO + 3\ R\text{-}C_6H_3O_2 + 6\ H^{+} \tag{1}$$

The reaction indicates that a polyphenol is oxidized to quinone, this transformation allows the reduction of $FeCl_3$ to FeO and Fe° (Mohan Kumar et al., 2013), so it is deduced that the nanoparticles obtained in the research would be formed by Fe°, FeO (characteristic black colour) with the presence of polyphenols in an acidic medium (Mystrioti et al., 2016). It is corroborated by other studies where they used Eucalyptus Globulus (Andrade-Zavaleta et al., 2022), tea (Xiao et al., 2020), Moringa Olifeira (Gautam et al., 2020b); olive (Essien et al., 2018), which reported the same constituents.

2.4 Characterization of iron nanoparticles

The characterization of the iron (Fe) nanoparticles biosynthesized with quinoa leaf extract using the above procedure, in terms of size, was performed by transmission electron microscopy (TEM). A Thermo Scientific Talos f200i microscope was used, with a working voltage of 200 kV. For this purpose, the Fe nanoparticle sample to be characterized was dispersed in an ethyl alcohol solution. A microdroplet was taken and placed in the aforementioned equipment on a copper grid support.
The composition and crystalline structure of the nanoparticles were characterized by fluorescence and X-ray diffraction in the 2 Θ range, respectively, using a Rigaku Miniflex 600 X-ray diffractometer equipped with a radiation source with a 40 kV/15 mA setting. The functional groups present in the extract and the iron nanoparticles were evaluated by Fourier transform infrared spectra (FTIR) in the range of 4000-650 cm^{-1} with a Perkin Elmer brand spectrophotometer, Frontier FTIR/NIR model.

3. Results

3.1 Identification of phenolic compounds in quinoa leaves

FTIR spectra were used to identify phenolic compounds in quinoa leaves that acted as reducers of $FeCl_3$ and formed iron nanoparticles (NpFe); the results are shown in Figure 3. It can be observed that it records wavelength peaks of 3341.49 cm^{-1} which identify the presence of hydroxyl groups OH- found in the phenolic compounds of the extract; there is also a sharp peak at 2983.19 cm^{-1} which indicates vibrations due to the presence of C-H groups from CH_2 and CH groups, indicative of the presence of an aromatic ring (Ar-H) in the extract (Somchaidee and Tedsree, 2018).
The same graph shows peaks at 1638.38 cm^{-1} related to C-N stretching vibrations of secondary amines (Pavan Kumar et al., 2020). There are spectra in the bands of 1412.97 and 1308.94 cm^{-1} that correspond to the bending of OH- groups, as well as in the stretching bands at 1044.90 and 877.07 cm^{-1} due to the vibrational stretching of the C-O group of polyols (Garole et al., 2018).
In this way, FTIR spectroscopy confirmed the presence of phenolic compounds in the quinoa leaf extract, which are reducing agents and stabilizers of ferric chloride ($FeCl_3$) for the biosynthesis of iron nanoparticles; this was corroborated by Andrade-Zavaleta et al. (2022), who confirmed the reducing action of phenolic compounds from eucalyptus leaves to form iron oxide (FeO) nanoparticles. Awwad and Salem (2013) also assert in a study that phenolic functional groups are responsible for the protection and stabilization of nanoparticles.

3.2 Identification of phenolic functional groups in iron nanoparticles (NpFe)

In the reduction process of iron chloride (0.1 M $FeCl_3$) with phenolic extract of quinoa leaves, the color of the solution changed from yellow to black, thus confirming the formation of iron nanoparticles (NpFe) (Abdelfatah et al., (2021); this occurs due to the complexation of Fe+3 with the phenolic groups present in the plant extract of quinoa leaves, when the interaction occurs in a metal-ligand form through the C=O bond. It should be noted that, in the reduction process for the formation of NpFe, there was a decrease in pH from 5 to 2.14, due to the generation of H^+ in the process when phenols were added. The iron nanoparticles (NpFe) were subjected to FTIR analysis to identify the functional groups present coating the nanoadsorbents. The results presented in the spectrum (see Figure 4), it is observed that, in the flat bands at 3227.46 cm^{-1} correspond to stretching vibrations of hydroxyl radicals (OH^-), characteristic of the presence of alcoholic and phenolic groups (Somchaidee and Tedsree, 2018); according to Vázquez-Guerrero et al. (2021), the presence of OH- radicals on the external surface of the nanoparticle allows the adsorption of metals on it. The absorption peaks between 1396.3 cm^{-1} and 1563.09 cm^{-1} would correspond to the stretching vibration of the carboxyl groups C=O, C-O, and COO-. These results are similar to those reported by Önal et al. (2019), whose results suggest the presence of the aforementioned functional groups.
Overall, the IR spectra showed that the Fe nanoparticles are coated with different functional groups from the plant extract, which contributes to the formation of covalent bonds that stabilize the nanoparticle and accelerate the absorption of the Fe-Crystalline nanoparticles. adsorption processes, because they provide interaction sites on the nanoparticle surface (Pavan Kumar et al., 2020). Likewise, it is also established that the band located at 1351.4 cm^{-1} is due to C-N stretching or OH- bending vibrations (Gautam et al., 2020). Likewise, it is observed

and predicted that the stretching vibration of carboxylic acids, ketones and esters is present in peaks from 1275.4 cm^{-1} to 1028.42 cm^{-1}, while the absorption peaks at 866.97 cm-1 and 789.64 cm^{-1} are attributed to the stretching vibration of the asymmetric C-O bond of phenolic groups and OH^- bond (Somchaidee and Tedsree, 2018).

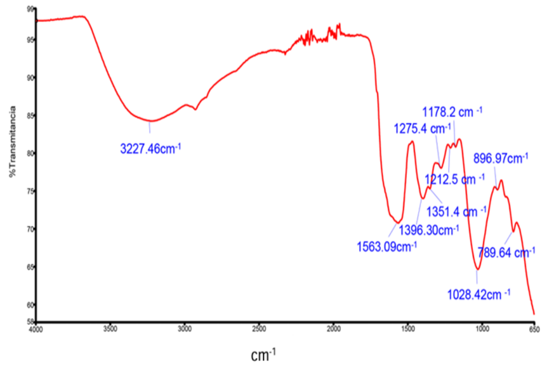

Figure 3: Functional groups determined by FTIR in the quinoa leaf extract

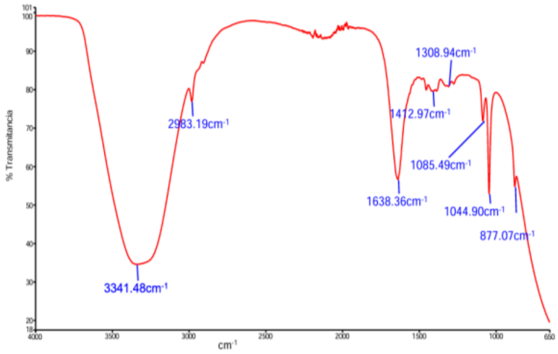

Figure 4: Functional groups determined by FTIR on the surface of Fe nanoparticles

3.4 Characterization of iron nanoparticles (NpFe)

The iron nanoparticles were characterized using Transmission Electron Microscopy (TEM) to determine the morphology (size and shape) of the nanoparticles, as recommended by the scientific literature (M. Gupta et al., 2020). In Figure 5 a,b,c, heterogeneous particles of sizes between 1-100 nm are observed, with ordered and rough shapes (Figure 5c) attributable to crystalline Fe (Arancibia-Miranda et al., 2014; Yang et al., 2019), as well as the presence of degenerated amorphous iron oxide and hydroxide (Giasuddin et al., 2007; Xi et al., 2010), respectively. Abdelfatah et al. (2021) suggest that the heterogeneity of the particles would provide information about their high surface area and reactivity towards contaminants and would facilitate active sites for physical exchanges and chemical reactions (Pavan Kumar et al., 2020).

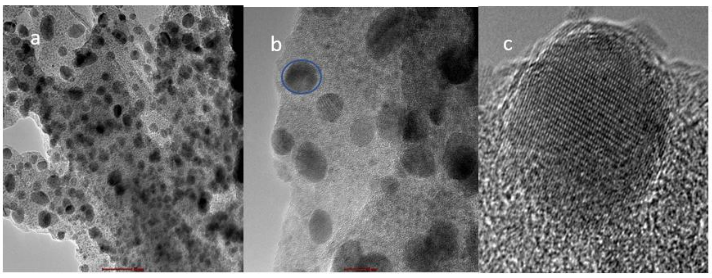

Figure 5: Micrographs of Fe nanoparticles biosynthesized with quinoa leaves

The images in Figure 5 show agglomerated nanoparticles, which give a circular shape and a larger size (Figure 5b). This agglomeration of nanoparticles occurs due to the variety of polyphenols present in the plant extracts that are involved in the research matrix (Ebrahiminezhad et al., 2017). This agglomeration happens due to the magnetic and electrostatic interaction between the functional groups present in the plant extract and the Fe (Yang et al., 2019). The results obtained are consistent with those reported by Önal et al., (2019) and Andrade-Zavaleta et al., (2022), who claim to have observed agglomerations in the obtained nanoparticles and infer that they may possibly be linked to organic stabilizing agents, which would be related to the organic functions observed in the FTIR. In short, the biosynthesized nanoparticles with plant extract exhibit heterogeneous morphology, a characteristic that facilitates the sorption processes as it presents active sites for physical and chemical exchanges (Pavan Kumar et al., 2020).

3.5 Composition percentages of iron nanoparticle constituents

The black color of the nanoparticles would indicate the presence of iron oxides (Kumar et al., 2015; Mystrioti et al., 2016) and their chemical composition was determined by X-ray fluorescence. The results showed particles with 19.4% Fe and 74.4% O, which, according to the scientific literature, correspond to Fe_2O_3 in hydrated form and with the presence of other components, due to the characteristic polymorphism of iron oxide (Tucek et al., 2012). In the research by Garole et al. (2018), percentages of 78.9% and 21.1% are indicated for Fe and O, respectively, and they infer that these percentages are close to $Fe2O_3$. The results show the amorphous nature (see Figure 6), which would be related to the matrix of the polyphenol extract that supports the nanoparticles (Giasuddin et al., 2007), observed in Figure 5a, which prevents visualizing the crystallinity of the nanoparticles. These results agree with those reported by Shahwan et al. (2011), who also found amorphous structures in iron nanoparticles synthesized with tea and Moringa Olifeira (Vázquez-Guerrero et al., 2021).

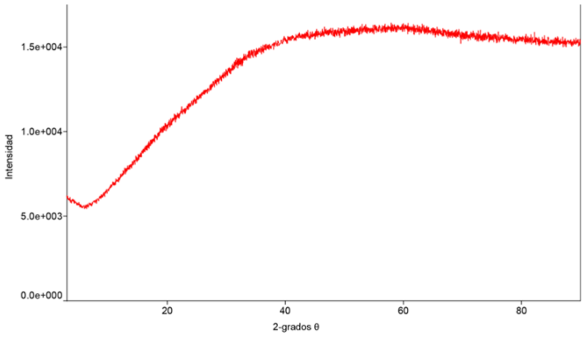

Figure 6. X-ray diffraction of Fe nanoparticles

4. Conclusion

Fe nanoparticles were biosynthesized with quinoa leaf extract, their characterization by TEM confirmed the presence of nanoparticles with a diameter of 1 to 100 nm, X-ray fluorescence showed that they were made up of 19.4 % Fe and 74.4 % O; X-ray diffraction also indicated the amorphous nature of the nanoparticles and FTIR allowed to identify the presence of phenolic groups in the structure of the nanoparticles, which act as stabilizing and protective agents for the nanoparticles, giving them special characteristics such as adsorption by electrostatic interaction.

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