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Synthesis of magnetic iron nanoparticles using *Zea mays* as a stabilizing agent

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Abstract

The objective of this research is to determine how to obtain magnetic iron nanoparticles through the coprecipitation technique using FeCl₃ and FeSO₄ as raw materials in an aqueous extract of purple corn (*Zea mays* L) as a stabilizing state. In recent decades, the manufacture and use of magnetic iron nanoparticles has created great importance and expectation due to their various applications in different areas, among which stands out their ability to eliminate waste that pollutes the environment. Because research design is experimental in nature due to the different samples carried out in laboratories. The selection of corn was randomized due to the ease of access to the product from a group of farmers who were previously trained to plant, cultivate, harvest, and dry purple corn. The corn antioxidants were extracted with boiling water, and the synthesis of the nanoparticles was performed by mixing FeCl₃, FeSO₄ in the extract solution at a temperature of 25°C for 30 min with agitation at a pH above 11 with the addition of NaOH. Characterization was performed by UV-Vis spectrophotometer. The coprecipitation method produced the magnetic iron nanoparticles using FeCl₃ and FeSO₄ as reagents and the aqueous extract of purple corn. The practical implications that this research will have are diverse, as theoretically it will be possible to carry out experiments with other types of corn to see the different results, as well as to use it to carry out different tests in places highly contaminated with heavy metals.

Keywords: Aqueous extract, Coprecipitation, Nanoparticles, Purple corn, Stabilizing agent, Magnetic iron nanoparticles.

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Transparency: The authors confirm that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

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1. Introduction

The nanometer scale provides the ability to analyze and understand matter from an atomic-molecular point of view. In recent times, nanometer-sized particles have generated great interest due to their electronic, spectral, and attractive characteristics [1]. Among the nanoparticles that commonly contain magnetic elements are iron, nickel, cobalt, and certain chemical compounds, with sizes smaller than a micrometer in diameter [2]. The uses of these materials are present in medicine, biology, physics, and chemistry, even have applications in technological fields and industry [3].

There is a wide range of nanoparticles with attractive properties, such as magnetite (Fe_3O_4), which has stood out as a promising alternative due to its biocompatibility and magnetism. It is a black mineral containing both iron +2 and iron +3, [4]. Fe^{2+} ions fill the centric places and vertices of a cube, while Fe^{3+} ions fill the octahedral spaces of a tetrahedron [5]. There are many chemical methods to elaborate magnetite nanoparticles with similar properties and size; among them are: thermal decomposition, hydrothermal synthesis, coprecipitation, and oxidation of iron hydroxide with KNO_3 [6]. Coprecipitation is possibly the most used technique because of its ease and chemical efficiency for the elaboration of iron nanoparticles [7]. Iron oxides (Fe_3O_4 or Fe_2O_3) are usually obtained by mixing stoichiometric amounts of ferrous and ferric compounds in solution [8].

A problem related to the properties of ferrofluids, is their surface area/volume ratio, which is agglutinated to decrease the surface energy due to the strong magnetic dipole-dipole attraction [9]. Further, the properties respond to the type of salts used, the ratio between Fe^{+2} and Fe^{+3} , temperature, pH, and the ionic concentration of the solution in which the reaction is carried out [10].

Any colloidal dispersion experiences a spontaneous process known as aging over time, which involves several aspects. First, the solid particles become more soluble in the liquid, which increases their primary size. Second, the colloidal particles adhere to each other through Van der Waals interactions, especially the magnetic contribution of magnetite, resulting in agglomeration. Thirdly, magnetite (Fe_3O_4) can be chemically transformed into maghemite (Fe_2O_3) by adsorption of molecular oxygen. Thus, covering the surface area of nanoparticles prevents the adhesion of particles that collide in the thermal movement provoked in the synthesis. In aqueous media, stabilized dispersions can be obtained by electrostatic repulsions [11] by positively or negatively charging the particles by applying acidic or basic solutions and applying layers of electrostatic steric stabilization to prevent nanoparticle clustering, which will help to have more surface area, due to less agglomeration and are effective in the separation of contaminants in sewage [12].

The time and speed of mixing intervene in the reaction favorably as in the yield (85 to 94%), size (23 ± 13 to 7 ± 4 nm), the agglutination of nanoparticles, which is influenced by the level of acidity or alkalinity in which the reaction occurs, obtaining acceptable nanoparticles for environmental applications [13]. Fe_3O_4 nanoparticles are characterized by different techniques, such as UV-Visible, FT-IR, XRD, SEM, and EDAX, whose UV-Vis spectra revealed an absorption maximum from 275 to 301 nm [14].

The characterization of nanoparticles from iron salts is carried out by UV-Vis spectroscopy, which, after analysis, exhibit maximum absorption peaks at 294 nm [15]. According to Sandhya and Kalaiselvam the extremely small samples of iron oxides can absorb and scatter UV light in the range of 330 nm to 450 nm using a non-edible seed coat of *borassus flabellifer* [16].

Green synthesis is an economically efficient, ecological, and socially sustainable alternative that seeks to reduce the use and production of hazardous substances. There are alternatives that contain the use of biocompatible predecessors such as biopolymers, amino acids, and sugars. In the production of these metallic nanomaterials, plant biomass is used as a precursor or synthesis additive that is the simplest alternative, with efficient and reproducible costs [17], whose products are natural, contain antioxidant properties, and participate in the reduction of metals [18] contained in plant cells [19] or in aqueous extracts of plants, which usually present active components [20]. In this sense, plant extracts have been used in *in vitro* processes in recent years to obtain nanoparticles, which are used for the bioreduction of ionic metals [21]. The phytochemicals existing in the extracts as reducing and stabilizing agents include flavonoids, phenolic compounds, cardiac glycosides, proteins, and sugars [15].

Purple corn is a grain native to America that contains anthocyanins and phenolic compounds that act as an antioxidant and natural stabilizer [22]. It has been reported that the chemical groups involved in the process of nanoparticle production are: alcohols, ketones, aldehydes, amines, carboxyls, hydroxyl groups, and sulfhydryls [23]. Therefore, we could use biological compounds containing these chemical groups to convert metal ions into nanoparticles. However, terpenes, heterocyclic flavonoids, polyphenolic compounds, reducing sugars, glutathione, and ascorbate are directly involved in their obtaining; therefore, proteins act as stabilizers, creating an organic layer by adsorption called corona [24].

1.1. Applications

There are spectrophotometric techniques that are performed to synthesize silver nanoparticles through redox reactions with reducing agents, oxidizing agents, and stabilizing agents that resulted satisfactorily and were proposed as an ecological alternative with economic costs [25]. The synthesis of nanoparticles biogenic technique is also an alternative that reduces toxicity and enhances activities against biological contamination. This whole process is thanks to the fungus (*Trichoderma harzianum*) that acts as a stabilizing agent [26]. In the same way the green synthesis Fe_3O_4 manages to have effects to behave as a magnetic agent [27], also existing the extracts of *Cleistocalyx operculatus* buds manage to extract in nanoparticles some contaminant minerals, resulting in very ecological and economic results in comparison with other experiments performed in prestigious laboratories [28], and it is so that there are experiments with different types of plants, ways of extraction, methods, and others to be able to extract minerals from contaminated sites near agricultural areas [29].

In many countries, the only motives and purposes for research are to find electrochemical and physical changes in different types of nanoparticles of the contaminating elements, for which plant extracts are being used as a fast, efficient, and above all, economical method [30]. Polymers from plastic water bottles are a potential threat to the world. This is due to the fact that they are harmful to health, so research continues on how to deal with this pollutant [31]. But papaya extract as a toxicity-reducing agent holds promise and is potentially biomedical that will succeed in sustaining the environment and humans [32]. The green synthesis approach, which uses plant extracts, is being used in many research studies because they are eco-friendly and safe for agriculture and human health [33].

The process of using nanoparticles is part of the solution techniques in the quest to conserve the environment without toxic waste and a promising ecology for the conservation of all living things; moreover, the extracts that are used are having good results as reducing agents [34]. Nanoparticles with different metals combat bacterial infections with a broad spectrum, despite the existence of resistance to some antibiotics, which is why saponin (AgNPs-S) has been used, which has been obtained from Ajwa dates (*Phoenix dactylifera* L.) as a reducing agent [35]. Other nanoparticles are used as catalytic agents, using *Vitisvinifera* L. leaves as secondary metabolic agents [36], and other nanoparticles are found in antibacterial-type hydrogels [37].

1.2. Justification and Objectives of the Research

Frequently, the synthesis of magnetic nanoparticles implies a high economic and energetic cost due to the necessary conditions and equipment, so this research has the general objective of synthesizing iron magnetic nanoparticles by the chemical coprecipitation method, starting with FeCl_3 and FeSO_4 as precursor salts, in the presence of an aqueous extract of purple corn (*Zea mays* L.) as a stabilizer. This technique is relatively easy, effective, environmentally friendly, and economically accessible.

2. Methodology

2.1. Materials and Methods

The research work follows an experimental design, involving several laboratory analyses to obtain the results. The samples of the main ingredient (purple corn) were collected by non-probabilistic and intentional sampling, due to the ease and access to the raw material. The cultivation of purple corn was carried out in the province of Cusco (the province of Cusco is located at an altitude of 3399 meters above sea level). This product was selected from a group of farmers who received training for planting and cultivation without the use of fungicides, where they were recommended to use only natural fertilizers. Once harvested, the product (purple corn) was also recommended to be dried naturally, i.e., in an open extension with normal ventilation and at room temperature. The corn was found and selected in ten stalls of the central market of the city of Cusco, called "San Pedro," in the month of September 2022.

The corn samples collected were heterogeneous due to the care taken in the selection and the previous classification and manual cleaning in order to separate damaged kernels, branches, or impurities that could interfere later in the experiment in the laboratories. Subsequently, the selected sample was moistened and then subjected to drying at a laboratory temperature of 105°C for 5 hours. The dried samples were ground until no residue remained, which was sieved with a No. 40 mesh, in order to obtain a fine, homogeneous sample with less surface area in contact with the solvent during extraction.

To obtain the purple corn extract containing the antioxidants, approximately 100 g of sample were placed in a balloon, which was boiled with pure and disinfected water at 85°C for 5 minutes, then cooled to room temperature, and finally passed through a filter to obtain the dark purple extract.

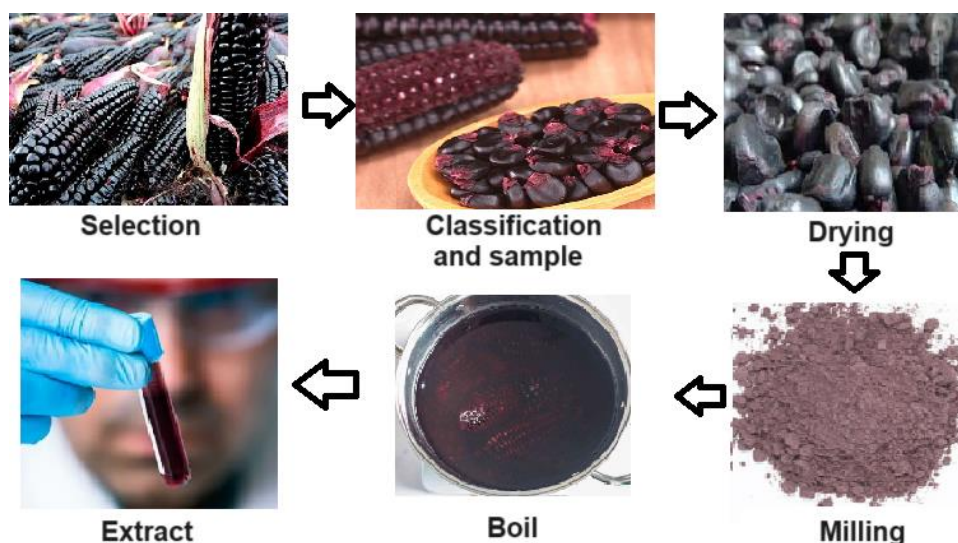


Figure 1.
Flow diagram for obtaining the corn extract.

We synthesized magnetic nanoparticles from ferric chloride and ferrous sulfate, in the following steps: 10ml of aqueous extract of purple corn was added to 10ml of 0.1M ferric chloride and 0.05M ferrous sulfate with agitation, heating the mixture to 25°C, and then 0.1M sodium hydroxide was added with agitation at 1000 rpm until reaching a pH of 11. It was left to stand for 30 minutes. After being filtered, the solid obtained was washed with a 50% hydroalcoholic solution to eliminate residual chlorides. The chloride-free solids were subjected to 500°C in a muffle for 3 hours, and finally, once cooled, the solid was subjected to a sonicator (JIOTECH, Model: EE-UU-05P) for 15 minutes at 60Hz and 20°C.

We carried out the solid's characterization in a UV-Vis spectrophotometer (THERMO, Model: EVOLUTION 300), adding 10 mg of sample and enough solvent to fill 3 ml.

3. Results

The aqueous extract of purple corn obtained has a dark purple coloration and reveals the presence of flavonoids through the Shinoda test, which gives an orange coloration, and the exposure to UV light, which shows a yellow-green coloration.

The nanoparticles obtained are brown-yellow in color with an earthy aspect, and to eliminate the presence of chlorides, product of the synthesis carried out, a 0.1M silver nitrate solution was used. The nanoparticles obtained when subjected to a magnetic field react favorably and the solids obtained are agglutinated, which makes it difficult to read in the UV-Vis spectrophotometer for their spectrometric characterization. To overcome this aspect, we proceeded to sonify at a frequency of 60Hz for 15 minutes.

Figure 2 shows the UV-Vis spectrum corresponding to the magnetic nanoparticles synthesized from FeCl₃ and FeSO₄ with significant peaks at 315nm and 361nm proteins that create an organic layer called corona through the adsorption process.

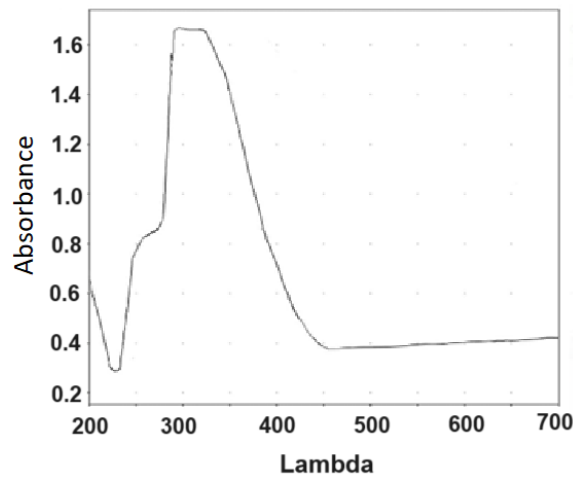


Figure 2. UV-Vis spectra of the magnetic nanoparticles synthesized from FeCl₃ and FeSO₄.

Figure 3 shows the results obtained in other experiments, and when compared, they have similar characteristics, so the research should continue to be subjected to more sophisticated instruments in order to maximize the obtaining of nanoparticles.

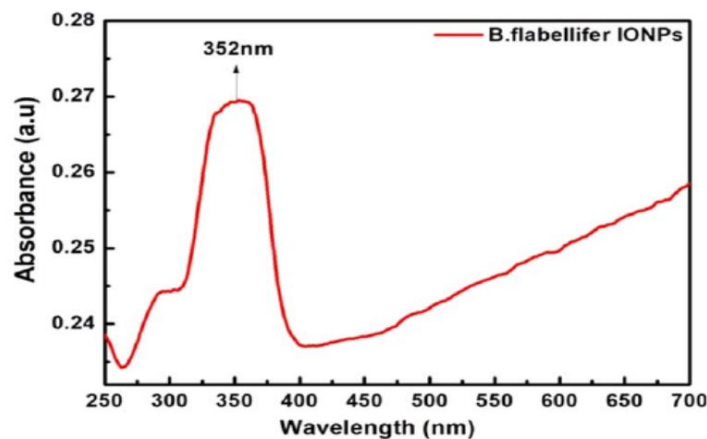


Figure 3. UV-vis spectrum of iron oxide nanoparticles.

4. Discussion

The purple corn extract, as in all the research, was proceeded according to the flowchart presented in [Figure 1](#), which presents antioxidants (in our case), which corroborates with what is mentioned by other authors at the international level, such as [Guillén-Sánchez, et al. \[22\]](#). The other studies of this type of methodology affirm that the extract of purple corn is a natural stabilizer, since it contains hydroxyl groups [\[23\]](#) and also contains proteins that form organic layers called corona through the adsorption process in obtaining nanoparticles that act as antioxidant agents [\[24\]](#).

Aqueous extracts of many plants usually contain one or more active components that release their properties [\[20\]](#) to be used in the bioreduction of metal ions, which are formed as nanoparticles [\[21\]](#). Flavonoids, phenolic compounds, cardiac glycosides, proteins, and sugars are also present in green extracts [\[15\]](#) and act as reducing and stabilizing agents [\[15\]](#). This alternative is economically efficient, environmentally, and socially sustainable and seeks to reduce the use and generation of hazardous substances for the health of living beings. The production of metallic nanomaterials using plant biomass as a precursor or additive for green synthesis is simpler, more cost-effective and more efficient [\[17\]](#). In addition, in vitro approaches using extracts of different plants in the bioreduction of ionic metals in contaminated sites such as rivers, operating mines, abandoned mines, agricultural centers, and other places have been developed in recent years of research [\[21\]](#).

The magnetic nanoparticles synthesized were from ferric chloride and ferrous sulfate by coprecipitation. There is a wide range of magnetic nanoparticles [\[4\]](#). Magnetite (Fe_3O_4), for example, has emerged as a promising alternative for decontamination due to its biocompatibility and excellent magnetic properties [\[5\]](#). Magnetite nanoparticles have similar properties (physical, chemical, and biological) and sizes, obtained by coprecipitation [\[6\]](#). Coprecipitation is possibly the most widely used technique due to its ease and chemical efficiency for the fabrication of iron nanoparticles [\[7\]](#). Usually we prepare iron oxides (Fe_3O_4 or Fe_2O_3) by mixing large stoichiometric amounts of ferrous and ferric salts in aqueous media [\[8\]](#).

The nanoparticles obtained with the different methods are brown-yellow in color with an earthy appearance. To eliminate the presence of chlorides resulting from the green synthesis, deionized water was used, which was tested with silver nitrate solution [\[25\]](#). The nanoparticles obtained when subjected to a magnetic field reacted favorably and the obtained solids were agglutinated, which made difficult the reading in the UV-Vis spectrophotometer difficult for its spectrometric characterization. To overcome this aspect, we proceeded to sonify at a frequency of 60Hz for 15 minutes. Nanometer-sized particles have created great expectations due to their electronic, magnetic properties and their behaviors to light [\[1\]](#). Nanoparticles that usually have magnetic elements are iron, with sizes less than one micrometer in diameter [\[2\]](#), which are used in medicine and biology, undoubtedly passing through physics and chemistry, even being applied in technological fields and in the reforestation industry [\[3\]](#).

A problem related to the properties of the ferrofluid is that they are nanoparticles with surface area/volume ratio that can agglutinate and decrease the surface energy due to the strong magnetic dipole-dipole attraction [\[9\]](#). Also, the physical chemical properties respond to the type of salts used, the ratio of Fe^{+2} and Fe^{+3} , the values of temperature, pH, and the concentration of ions present in the solution in which the reaction is carried out, which have positive effects for biological preservation [\[10\]](#).

In any colloidal dispersion, with time, a spontaneous process known as aging occurs. Therefore, coating the surface of the nanoparticles is a way to avoid the adhesion of the particles that collide in the thermal movement produced in the synthesis. Aqueous extract of purple corn was used as a stabilizing medium to thus avoid the agglutination of the nanoparticles obtained. In an aqueous medium, the electrostatic and spherical stabilization layers prevented the nanoparticles from agglomerating [\[11\]](#), which will help to possess larger surface areas, and due to less agglomeration, these will be more effective in the removal of pollutants in wastewater [\[12\]](#).

The speed and time of mixing always intervene favorably in the production, with 85 to 94 %, of the nanoparticle size ranging from 23 ± 13 to 7 ± 4 nm. The acidity or alkalinity intervenes in the agglutination, obtaining nanoparticles with acceptable sizes for environmental uses [\[13\]](#).

The Fe_3O_4 nanoparticles are characterized by different techniques, such as UV-Visible, FT-IR, XRD, SEM, and EDAX, whose UV-Vis spectra revealed an absorption maximum from 275 to 301 nm [\[14\]](#). Also the characterization of iron salt nanoparticles was found. UV-Vis spectroscopy showed peaks with absorption maxima at 294 nm [\[15\]](#) and also showed a UV absorption zone between 330 nm and 450 nm due to the absorption and scattering of light by the nanoparticles [\[16\]](#).

Many nanoparticles showed that in their residual growth they are potent inhibitors and no agricultural effects were obtained, concluding that nanoparticles are essential factors to have biological control and achieve great sustainability in agriculture [\[26\]](#), so Lawsonialnermis extract is used, which proved to be a reducing and stabilizing agent, and above all, it is natural and ecological [\[30\]](#). The extracts are made with plants, where the bark of the trees, wet or dry roots, leaves, fruits or seeds are used, with which different experiments are performed and then studied and analyzed in nanoparticles for the absorption of some contaminant metals or as an option for water decontamination [\[33\]](#), in which it has been demonstrated that AgNPs-S has antioxidant activities and provides information on the potential of nanoparticles, especially if they are therapeutic agents that manage to develop antibiotics [\[35\]](#).

5. Conclusion

Magnetic iron nanoparticles were synthesized by chemical coprecipitation method starting from FeCl_3 and FeSO_4 in the presence of an aqueous extract of purple corn, whose magnetic property was verified through UV-Vis spectrophotometry showing characteristic peaks of iron oxide nanoparticles with magnetic capacity.

If purple corn served as a stabilizer, then there must be other products in the area with which tests should be carried out and results compared to optimize the greatest amount of magnetic iron nanoparticles, remembering that in Peru there is a great variety of products similar to corn.

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