

Characterisation of Green-Synthesized Fe₃O₄ Nanoparticles Using Areca Nut Seed Extract (*Areca catechu* L.)

Shella Garelita, Lucky Zaehir Maulana, Febri Berthalita Pujaningsih, Rista Mutia Anggraini, Frastica Deswardani*

Department of Physics, Faculty of Science and Technology, Universitas Jambi, Jambi, Indonesia

Abstract

Fe₃O₄ (magnetite) nanoparticles have attracted considerable attention due to their potential applications in various fields. In this study, Fe₃O₄ nanoparticles were synthesized through a green synthesis method using areca nut (*Areca catechu*) extract as a reducing and stabilizing agent, with varying concentrations of 50% (GSP-50%), 80% (GSP-80%), and 100% (GSP-100%). X-ray diffraction (XRD) analysis showed the presence of an iron oxide phase consistent with Fe₃O₄. The crystallite size decreased from 11.18 nm to 8.49 nm with increasing extract concentration, accompanied by an increase in the degree of crystallinity from 67.38% to 75.46%, while the lattice parameter remained relatively constant. Scanning Electron Microscopy (SEM) analysis revealed agglomerated particle morphology, indicating that the observed particle size corresponds to an aggregate structure rather than individual nanoparticles. UV-Vis spectroscopy showed a blue shift in absorption with increasing extract concentration. The estimated optical band gap ranged from 3.3 eV to 3.5 eV, which may be influenced by particle size effects and surface state. Overall, the concentration of areca nut extract significantly affected the structural and optical characteristics of the synthesized iron oxide nanoparticles.

Keywords: Nanoparticles, Fe₃O₄, Green synthesis, Iron sand, and *Areca catechu*.

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✉ Correspondence address:
Department of Physics, Faculty of Science and Technology, Jambi University
E-mail: frastica.deswardani@unja.ac.id

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INTRODUCTION

Nanomaterials have created a technological revolution that has led to the emergence of various nano-sized materials with many benefits in various fields. Nano-sized particles have become very popular because they have properties that are not found in macro particles in general (Sajid, 2022). The development of nanomaterials is evidenced by the existence of magnetic nanoparticles that have significant implications and practical applications (Hendrian & Munasir, 2023a). There are 16 types of iron oxides, among which Fe_3O_4 nanoparticles have attracted much interest from researchers (Rajendran, 2019). The synthesis of Fe_3O_4 nanoparticles offers various advantageous properties and applications in various fields such as adsorption and catalysis.

One type of iron oxide material that has been extensively developed is iron sand (Didik et al., 2020). The use of iron sand as a magnetic material has been widely researched and developed (Elsafitri et al., 2020). The darker the colour of the sand, the higher the iron mineral content (Bahfie et al., 2022). Iron sand has dominant constituent elements of iron (Fe) and oxygen (O_2). In the Batanghari River in Jambi Province, the existence of iron sand with Fe_3O_4 content is estimated to be quite abundant (Sinurat et al., 2021). The development of this innovation requires an appropriate synthesis method so that nanoparticles with controlled size and morphology can be obtained. The impact of using the nanoparticles Fe_3O_4 formation method needs to be considered.

The green synthesis method utilises extracts from natural materials to replace hazardous chemicals in an environmentally friendly manner (Watuna et al., 2025). The use of environmentally friendly green synthesis methods in the formation of Fe_3O_4 nanoparticles from iron sand can reduce the use of hazardous chemicals that cause environmental toxicity (Hendrian & Munasir, 2023b). Green synthesis with the addition of plant extracts can produce nanomaterials with highly stable properties in the right shape and size, as well as minimising the possibility of contamination (Okto & Munasir, 2023). Nanoparticle synthesis using plants is an alternative that is easy, simple, and currently developing. Plants contain various bioactive compounds that are useful for the synthesis and stabilisation of nanoparticles (Roy et al., 2023). In addition, the content of plants can also act as a reducing agent and help accelerate the synthesis process.

Areca nut is one of the largest commodities traded and exported in Jambi Province. Areca nuts in the Jambi region are of superior quality because they have a low water content of less than 6%. Areca nut seeds are the most widely used part because they contain various bioactive compounds (Kumala, 2024). Areca nut seeds contain several key chemical compounds, namely alkaloids, saponins, and flavonoids (Adi, 2007). In the process of forming Fe_3O_4 nanoparticles, the natural compounds in areca nut seeds can be used as reducing agents and stabilisers, replacing chemical substances.

Based on the potential described above, this study will synthesise Fe_3O_4 nanoparticles using areca nut extract (*Areca catechu* L.). The treatment that will be applied to obtain the desired results is through concentration variation. The concentration of the agent will affect the growth rate of the crystals that form the nanoparticles during synthesis (Septiani & Muldarisnur, 2022). The analysis of the synthesis results was obtained from X-Ray Diffraction (XRD) characterisation tests to identify nanoparticle characteristics such as crystal structure, SEM to observe the surface morphology of the material, and UV-VIS to obtain the band gap energy value of the magnetite nanoparticles produced.

METHOD

This research was conducted in three stages, the first stage involved the collection and preparation of iron sand, the second stage consisted of the extraction of areca nut using the maceration method, and the third stage focused on the synthesis of Fe_3O_4 nanoparticles from iron sand using areca nut extract. The purpose of this study was to investigate the effect of varying the concentration

of areca nut extract on the characteristics of the resulting nanoparticles Fe_3O_4 . The concentrations of areca nut extract used in this study were 50%, 80%, and 100%. The synthesized samples were then characterized using X-ray diffraction (XRD) and UV-Vis spectroscopy (UV-Vis). The equipment used in this study included a permanent magnet, sieves (60 mesh and 120 mesh), mortar, electric stove, magnetic stirrer, dropper pipette, measuring cylinder, beaker, rotary vacuum evaporator, filter paper, petri dish, and oven. The materials used consisted of iron sand taken from the Batanghari River, areca nut seeds, distilled water, 96% ethanol, hydrochloric acid (2.8 M HCl), and ammonium hydroxide (25% NH_4OH).

In the initial process, iron sand taken from the Batanghari River was dried in the sun. The dried sand was then sieved using a conventional sieve and separated from non-magnetic materials using a permanent magnet. The magnetic separation process was repeated 20 times until black iron sand was obtained. The separated iron sand was then sieved using a 120-mesh sieve and ground in a mortar to obtain finer particles. The fine iron sand was washed with tap water and rinsed twice with 250 mL of distilled water. After washing, the iron sand was dried in the sun for one day to determine the remaining moisture content after the washing process.

For the extraction process, areca nuts were obtained from Mendalo Darat Village, Muaro Jambi Regency. The areca nuts used in this study were ripe fruits characterized by yellow skin. The areca nuts were peeled and split to obtain intact, high-quality seeds. After drying until they shrank, the areca nuts were crushed, blended into a fine powder, and sieved using a 60-mesh sieve. A total of 400 g of areca nut powder was mixed with 96% ethanol at a 1:5 ratio, corresponding to a total solvent volume of 2 L (Jamilah et al., 2025). The mixture was stirred for 30 minutes using a magnetic stirrer, then covered with aluminum foil and stored in a dark place. The maceration process was repeated twice, with the 96% ethanol replaced every 24 hours. The extract was filtered using Whatman No. 1 filter paper to obtain the filtrate, which was then concentrated using a rotary vacuum evaporator at 55°C to produce a thick extract.

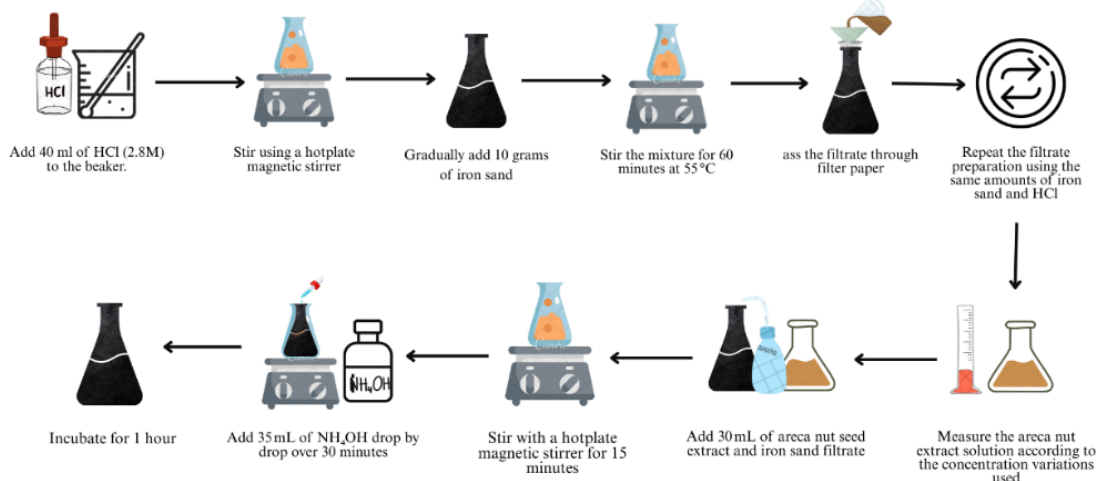


Figure 1. Production of magnetic nanoparticles with areca nut extract

After the iron sand and areca nut extract were obtained, the nanoparticle synthesis process was carried out. Ten grams of iron sand was dissolved in 40 mL of 2.8 M hydrochloric acid and stirred for 60 minutes at 55°C using a magnetic stirrer. The resulting solution was filtered using Whatman Grade 1 filter paper to obtain the filtrate. This procedure was repeated using 20 g of iron sand and 80 mL of 2.8 M hydrochloric acid for each experiment. The filtrate obtained was then measured to a volume of 30 mL, after which the areca nut extract was added according to the specified concentration.

The areca nut extract used in this study was prepared by dissolving the extract in distilled water at a 1:10 ratio relative to the mass of the areca nut extract. The extract was added at varying

concentrations of 50%, 80%, and 100%, designated as GSP (Green Synthesis Areca Nut). Each sample was stirred for 15 minutes at 60°C using a magnetic stirrer. Next, 35 mL of ammonium hydroxide (NH_4OH) was added dropwise to the solution for 30 minutes while maintaining the temperature at 60°C. The mixture was then allowed to stand for 60 minutes to facilitate precipitate formation. The resulting precipitate was washed seven times with distilled water until it reached a neutral pH (pH 7) (Fatimah et al., 2023). The precipitate was then separated from the solution using filter paper, resulting in a dense, black, sand-like precipitate. The collected sediment was left overnight and then dried in an oven at 100°C for 2 hours. This drying process was carried out to obtain a completely dry sediment in powder form with no residual water content.



Figure 2. Nanoparticle samples (a) Iron sand synthesis sample after precipitation and (b) Green synthesis results of Fe_3O_4 nanoparticles.

RESULTS AND DISCUSSION

Innovation in the development of magnetic nanoparticle materials using environmentally friendly systems continued to grow. This study aimed to examine the effect of variations in the concentration of areca nut seed extract on the formation of Fe_3O_4 nanoparticles. Three concentration variations were employed, namely 50% (GSP-50%), 80% (GSP-80%), and 100% (GSP-100%). The three powder samples produced were subsequently analyzed using several characterization techniques, including X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Ultraviolet-Visible (UV-Vis) spectroscopy.

X-Ray Diffraction (XRD) characterization results

Testing using XRD was conducted to obtain data on crystallite size, lattice parameters, and degree of crystallinity. Crystal identification was performed by comparing diffraction peaks to reference data of 2θ angle values from the Crystallography Open Database (COD). The reference data used corresponded to Fe_3O_4 (magnetite) with COD ID 1011032. The data were processed in the form of a diffractogram presented in Figure 3. The observed peaks indicated that the samples exhibited diffraction features consistent with magnetite like spinel iron oxide phases (Fe_3O_4). The diffraction patterns exhibited similar features, characterized by several sharp diffraction peaks, and there was consistency in the Miller indices (hkl) across the three samples.

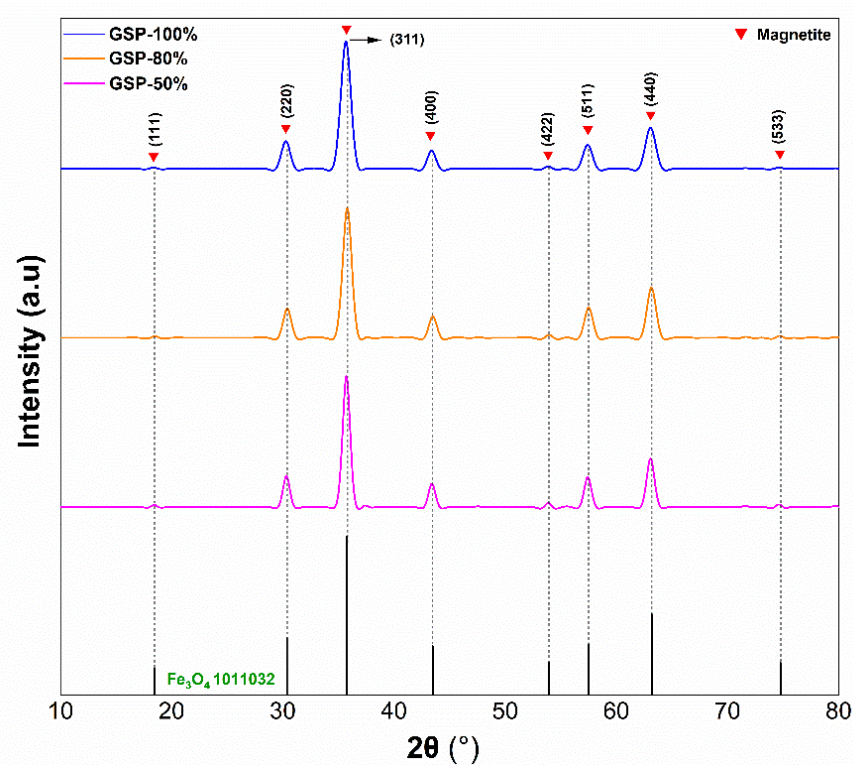


Figure 3. XRD Diffractogram of Fe₃O₄ synthesized with varying concentrations areca nut seed extract.

Based on Figure 3, a shift in the position of the diffraction peaks is visible in the three samples. This may occur due to the influence of the variation in the extract concentration value used in the synthesis process. Areca nut extract, as an active component, acts as a reducing and stabilizing agent to control crystal growth. Stronger interactions between bioactive compounds and the crystal surface occur at higher extract concentrations. This mechanism is related to the surface energy of the particles, which tends to increase and affects the growth and stability of the formed crystal phase (Prahesti & Munasir, 2025). This causes the lattice structure to stretch because more active components in the extract enter the surface of the crystal structure.

Data obtained from the diffractograms were used to determine the crystallite size and lattice parameters. The crystallite size was calculated using the Debye Scherrer equation in equation 1.

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

Where λ = wavelength, β = Full Width at Half Maximum (FWHM) which is the peak width at half intensity, K = crystal form factor constant, and θ = half the peak diffraction angle (Sari et al., 2021). Meanwhile, the lattice parameter (a) is determined using equation 2

$$a = d\sqrt{h^2 + k^2 + l^2} \quad (2)$$

Where d is the distance between crystal planes at angle θ and (hkl) is the Miller index value. The distance between the planes is determined using Bragg's law in equation 3.

$$n\lambda = 2d\sin\theta \quad (3)$$

Where the n value indicates the diffraction order (Febriliani et al., 2025).

Table 1. Lattice parameters and crystallite size of Fe₃O₄ calculated from the (311) XRD peak

Sample	2θ (°) ₍₃₁₁₎	d (Å) ₍₃₁₁₎	a (Å) ₍₃₁₁₎	D (nm) ₍₃₁₁₎
GSP-50%	35.71	2.51	8.32	11.18
GSP-80%	35.71	2.51	8.32	9.38

Table 1 displays the lattice parameters (a) and crystallite size (D), specifically at the (311) peak. The presence of the (311) diffraction peak shows a strong characteristic, marked by the highest intensity compared to other diffraction peaks. The decrease in crystallite size with increasing concentration indicates that the extract successfully formed a layer that functions as a capping agent, namely a compound that can slow particle growth so that it remains stable and does not clump (Bemis et al., 2023). High concentrations produce more capping agent, which causes the distance between crystal lattice planes and crystallite size to become smaller.

Table 2. Degree of crystallinity of Fe₃O₄ nanoparticles

Sample	Degree of Crystallinity
GSP-50%	67.38 %
GSP-80%	74.57 %
GSP-100%	75.46 %

The level of atomic order in a nanoparticle's crystal structure can be seen through its crystallinity. The calculation to determine the degree of crystallinity is as follows.

$$\%crystallinity = \frac{Crystalline\ fraction\ area}{Diffractogram\ area} \times 100 \quad (4)$$

The higher the percentage of crystallinity, the more regular and better the crystal structure of a nanoparticle. The results showed that samples with a concentration variation of 100% had the highest percentage of crystallinity, indicating that increasing the extract concentration can increase the level of crystallinity.

Scanning Electron Microscope (SEM) characterization results

The results of the Scanning Electron Microscope (SEM) characterization test were analyzed using ImageJ software. Figure 4 shows the SEM characterization results for three variations of extract concentration, namely GSP-50%, GSP-80%, and GSP-100%. The SEM image scale used was 5 μ m with a magnification of 10,000 times. In general, it can be seen that all three samples show the typical surface morphology of Fe₃O₄ nanoparticles with the formation of agglomerates. The surface texture still looks relatively rough with the presence of small particles attached to larger agglomerates. The agglomeration phenomenon is a common characteristic in the synthesis of Fe₃O₄ nanoparticles that occurs due to magnetic interactions between particles (Febriyani & Aini, 2023; Kurniasari et al., 2025).

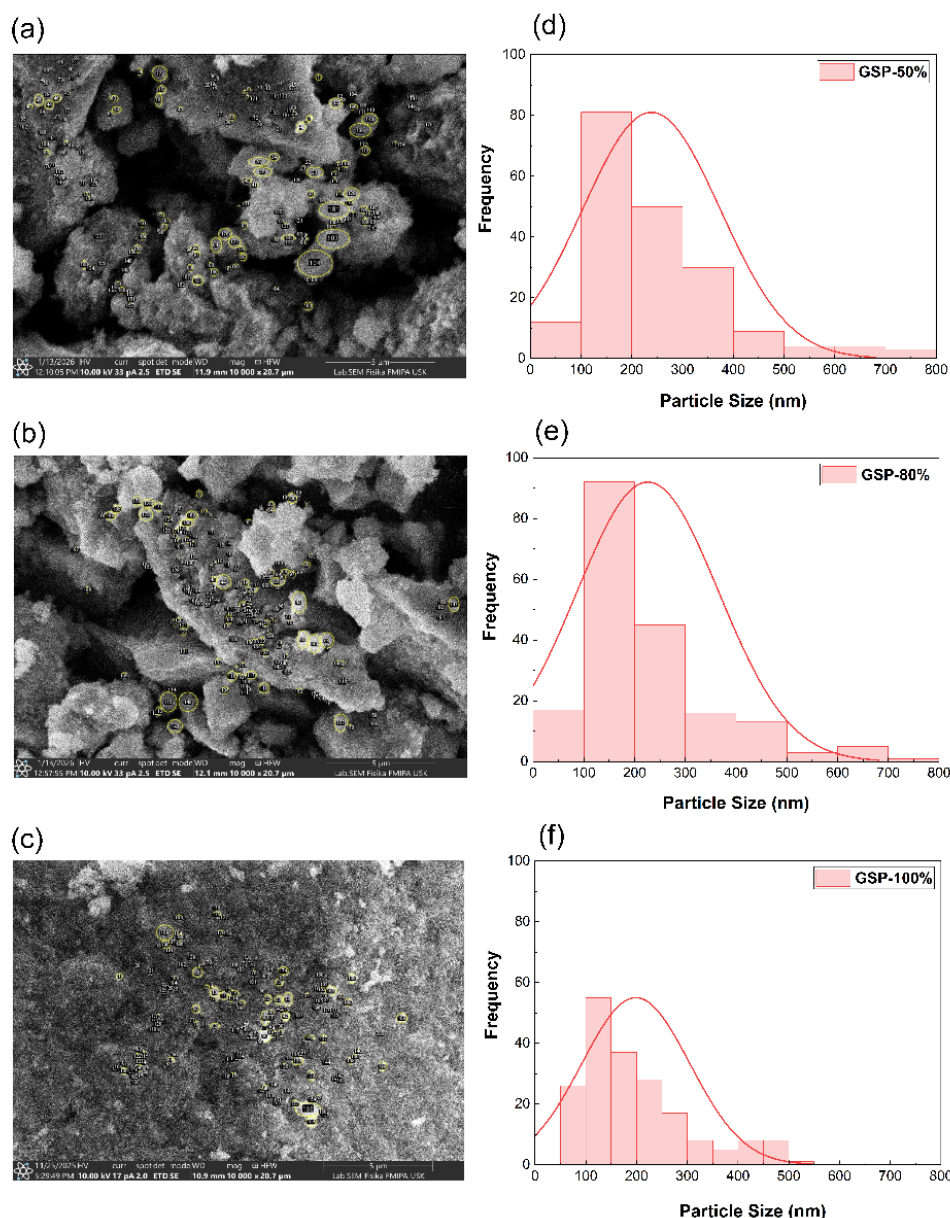


Figure 4. SEM morphology and particle size distribution of green-synthesized Fe_3O_4 nanoparticles at different extract concentrations: (a,d) GSP-50%, (b,e) GSP-80%, and (c,f) GSP-100%.

The quantitative distribution of the data can be more clearly observed in the histogram of Figure 4 (a-c). The histogram can analyze the distribution of particle size and the level of particle uniformity in the samples. Most of the particle sizes of the three samples decreased with increasing extract concentration. This was accompanied by a number of much finer and relatively more even particles. The results of the histogram analysis showed that the three samples had a relatively asymmetrical estimated particle size distribution, characterized by the presence of a tail distribution at larger particle sizes.

In the morphological variation of 50% concentration in Figure 4 (d), the particles form large, irregular clumps. This also occurs in the 80% concentration variation in Figure 4 (e). The estimated particle size is relatively smaller, but it is still difficult to measure individually because the particles have combined to form agglomerates with micrometer sizes. Meanwhile, in the 100% concentration variation in Figure 4 (f), the morphology shows much finer and relatively even particle sizes. However,

this occurs due to agglomeration that is much higher than the other two concentration variations. Smaller nanoparticles have higher surface free energy, this energy encourages small particles to combine into larger particles, resulting in particle clumping or agglomeration, one of the factors caused by concentration (Nasirpour et al., 2023).

Although the green synthesis process is capable of producing nano-sized particles, the particle size distribution is still not homogeneous. The particles form dense clumps that allow for an increase in effective surface area (Urmi et al., 2025). At the 100% concentration variation, the morphology displayed shows a much finer particle size. However, the resulting agglomeration is much higher than the other two concentration variations. Smaller nanoparticles have higher surface free energy, this energy encourages small particles to combine with larger particles, resulting in particle clumping or agglomeration, one of the factors caused by concentration (Nasirpour et al., 2023).

Tabel 3. Estimation of the mean particle size distribution

Sample	Particle size (nm)
GSP-50%	238 ± 135
GSP-80%	225 ± 139
GSP-100%	198 ± 106

Based on Table 6, there is a decrease in the average particle size value as the concentration variation increases. All observed particles are in the range of more than 200 nm. In Figure 4 (d-f), the particle size uniformity in samples with higher concentration variations such as GSP-100% has a narrower distribution. Meanwhile, in the concentration variations of GSP-50% and GSP-80%, the standard deviation value becomes larger, indicating heterogeneous particle size uniformity. This decrease indicates the influence of the concentration on particle shrinkage which simultaneously narrows the size distribution.

The relatively concentrated size distribution estimation at small sizes indicates that at 100% extract concentration, the role of areca nut seed extract as a reducing and stabilizing agent is quite effective in inhibiting excessive particle growth. When compared to the crystallite size obtained from XRD analysis, the particle size appears larger in the SEM image. The difference in size is caused by several factors such as the presence of agglomeration phenomena, where the observed particles are composed of several crystallites (Nengsih et al., 2023). Although the particle size seen in the SEM image appears larger, the structure is still composed of nanometer-sized particles.

UV-Vis characterization results

The use of UV-Vis characterization tests is carried out for energy band gap analysis which functions to see the material's ability to absorb light and photocatalytic activity. The band gap is the energy difference between the highest filled electron energy level (valence band) and the lowest empty electron energy level (conduction band) (Dwiriani et al., 2025). The energy band gap is divided into two, namely the direct band gap and the indirect band gap.

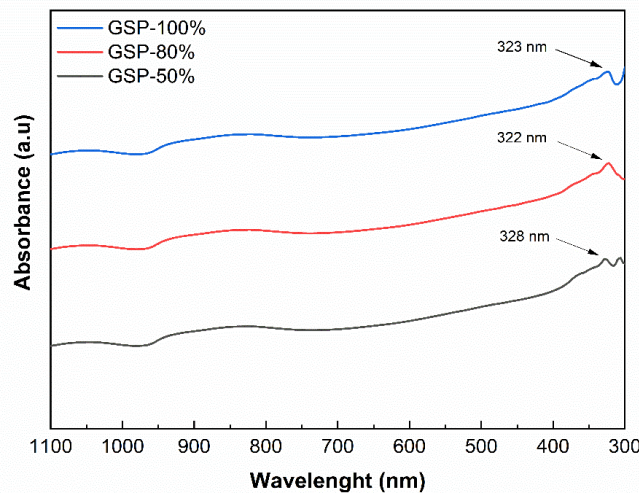


Figure 5. UV-Vis absorbance intensity spectra showing the optical response of Fe_3O_4 nanoparticles for samples GSP-50%, GSP-80%, and GSP-100%.

In the 100% GSP sample, the high concentration used significantly reduces the distance between molecules and alters their ability to absorb radiation at certain wavelengths. This results in a deviation phenomenon or a non-linear relationship between concentration volume and absorbance, (Kiteto & Mecha, 2025). Meanwhile, at a 50% concentration, interparticle interactions were relatively limited, leading to lower absorption. In contrast, at an 80% concentration, absorption was enhanced due to more effective interparticle interactions, resulting in improved particle dispersion.

To obtain the energy band gap value, the Tauc Plot method is used, which is shown in the following equation.

$$(\propto h\nu)^n = A(h\nu - E_g) \quad (5)$$

where $\propto$ is the absorption coefficient, h is the Planck constant, E_g is the optical band gap of the semiconductor, ν is the frequency, A is the proportionality constant, and n is the Tauc exponent. The Tauc coefficient has a value where $n = 2$ is used for indirect or direct transitions and $n = 1/2$ for direct or indirect transitions (Andrade et al., 2024).

In several previous research results, Fe_3O_4 nanoparticle materials have an energy band gap value in the range of 3-4 eV. The band gap of the synthesized Fe_3O_4 nanoparticles was greater than that of bluk magnetite (0.20 eV), which was attributes to the quantum confinement effect. The optical band gap of Fe_3O_4 nanoparticles had previously been reported to range from 3.23-2.9 eV (Mbuyazi & Ajibade, 2024). The relatively high band gap values were caused by quantum confinement, which occurred when the particle size became sufficiently small to alter the electronic properties of the material, thereby increasing the band gap energy. Crismeli et al. (2024) synthesized Fe_3O_4 nanoparticles with a band hap of 3.67 eV. Another study by Mbuyazi and Ajibade (2024) reported Fe_3O_4 nanoparticles with band gap values ranging from 3.27 to 3.53 eV. In the study, resulting energy band gap was comparable to those reported previously.

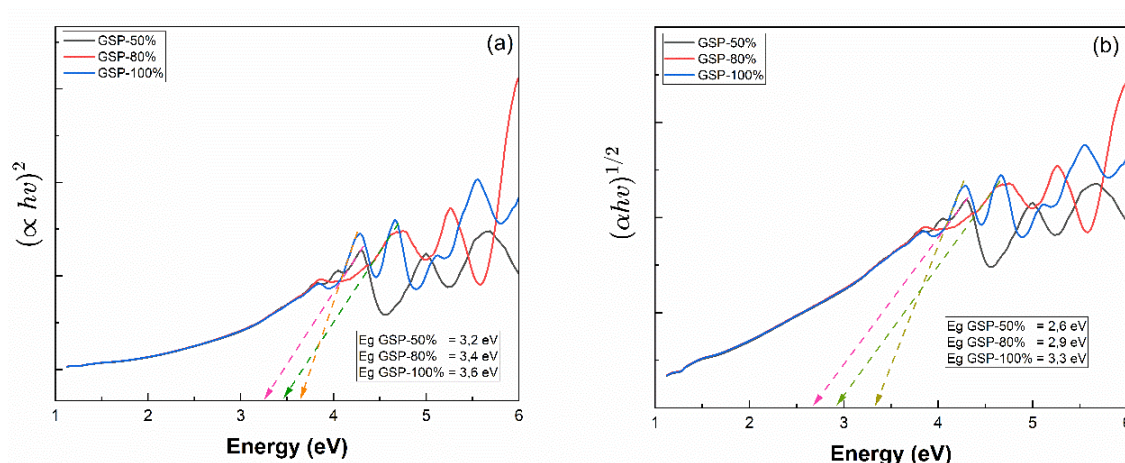


Figure 6. Determination of the energy band gap of Fe₃O₄ nanoparticles using Tauc plots: (a) direct transition and (b) indirect transition.

Based on the results shown in Figure 6, the energy band gap value in the direct transition (direct band gap) has a more regular linearity characteristic where there is an increase in the energy band gap along with increasing variations in the concentration used. In this study, the estimated optical transition energy was 3.3-3.5 eV, which may reflect nanoscale and surface-related effects in the synthesized nanoparticles. The UV-Vis data test results show that the energy band gap for the 100% concentration variation has a higher value than the other two variations. This value is consistent with the results obtained for previous nanoparticle sizes, which is in accordance with the theory that explains that reducing the size of the nanoparticle will increase the band gap energy. A decrease in the energy band gap will facilitate the transfer of electrons from the valence band to the conduction band, thereby affecting the absorbance intensity of the nanoparticles (Madden & Zwijnenburg, 2024). The energy value at a smaller band gap also represents a less perfect particle structure, so that electrons can move easily (Simsek et al., 2022).

CONCLUSION

The green synthesis of Fe₃O₄ nanoparticles from iron sand using areca nut extract was achieved. XRD analysis confirmed the formation of Fe₃O₄ (magnetite), with crystallite size decreasing as the extract concentration increased. The degree of crystallinity also increased indicating improvement in the crystal structure. SEM observations revealed that higher extract concentrations produced finer particles with a more uniform size distribution. However, an increased in extract concentration also resulted in higher particle agglomeration due to increased surface energy. UV-Vis analysis showed that the optical band gap increased up to 3.5 eV, indicating size dependent optical behavior of the nanoparticles. Overall, increasing the extract concentration enhanced crystallinity and reduces crystallite size, while simultaneously promoting particle agglomeration, highlighting a trade off between structural refinement and morphological stability. Among the investigated samples, GSP-100% exhibited the most favorable structural and optical properties, indicating that higher extract concentration plays a crucial role in controlling nanoparticle growth under the experimental condition.

DECLARATION OF GENERATIVE AI

The authors declare that no generative AI or AI-assisted technologies were used in the writing process of this manuscript.

CONFLICTING INTEREST STATEMENT

The authors states that there is no conflict of interest in the publication of this article.

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