MULTIDISCIPLINARY APPLIED RESEARCH AND INNOVATION
e-ISSN: 2773-4773

MARI

Vol. 6 No. 1 (2025) 105-111
https://publisher.uthm.edu.my/periodicals/index.php/mari

Sustainable Synthesis of Magnetic Nanoparticles Through Green Chemistry

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DOI: https://doi.org/10.30880/mari.2025.06.01.015

Article Info

Received: 01 October 2024
Accepted: 30 November 2024
Available online: 15 January 2025

Keywords

Iron nanoparticles, C*arica papaya*, magnetic, Fe3O4-NPs, MFeNPs, MNPs, XRD, EDS, FTIR

Abstract

The green synthesis of magnetic nanoparticles (MNPs) offers an environmentally friendly alternative to conventional chemical methods, utilising natural plant extracts as reducing and stabilising agents. This study reports the synthesis of iron oxide (Fe_3O_4) magnetic nanoparticles using extracts from different parts of *Carica papaya*, including leaves, branches, and peel. The phytochemicals present in these extracts acted as reducing agents to convert iron salts into iron oxide nanoparticles and provided stabilisation through capping. The synthesised nanoparticles were characterised by Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS) to determine their chemical, morphological, and elemental properties. FTIR spectra indicated functional groups from the *Carica papaya* extracts on the nanoparticle surface, confirming successful capping and stabilisation. SEM images revealed spherical nanoparticles with an average size of 25µm, demonstrating uniform distribution. EDS analysis confirmed the elemental composition, verifying the presence of iron and oxygen and indicating high purity of the Fe_3O_4 nanoparticles. The use of various plant parts not only resulted in a simple and cost-effective synthesis process but also enhanced the biocompatibility of the nanoparticles. The biogenic Fe_3O_4 nanoparticles demonstrated significant potential for applications in biomedicine, particularly in targeted drug delivery systems. This study underscores the importance of utilising renewable resources in nanomaterial production, contributing to sustainable and eco-friendly nanotechnology.

1. Introduction

Nanoparticles (NPs) are particulate materials with at least one dimension smaller than 100 nm, offering unique physicochemical properties that are valuable in various fields [1]. There is growing interest in the application of nanoscale magnetite particles in environmental studies due to their size, structure, and surface characteristics, which directly influence their chemical behavior and application [1]. The size, shape, and aggregation tendency of NPs significantly impact their functionality. Smaller NPs exhibit increased viscosity, melting point, thermal conductivity, and antimicrobial activity. Additionally, their reduced size facilitates intravenous administration of poorly soluble particles without obstructing blood vessels [2].

Recent advances in nanotechnology have broadened its use in diagnosing and treating diseases, particularly cancer [2]. Magnetite nanoparticles have been utilised for targeted drug delivery, hyperthermia treatment, and immunoassays [3]. The green synthesis of iron nanoparticles using biological extracts offers high stability and reduced toxicity, preventing aggregation and oxidation, making the process efficient, affordable, and environmentally friendly [2,4]. This method avoids the need for hazardous chemicals and high energy inputs.

Iron oxide nanomaterials have garnered significant attention for developing nanotherapeutic agents that combat free radicals, which are known to cause various diseases [4]. Phytochemical-Assisted Green Synthesis (Ph-ALE) enhances the biological applications of Fe3O4-NPs by utilising phytochemicals that efficiently contribute to nanoparticle synthesis.

Nanoparticles play a crucial role in water treatment due to their large functional surface area and magnetic properties, enabling applications in catalysis, biosensors, magnetic resonance, and wastewater treatment [5,6]. Various synthesis methods, including physical, chemical, and biological approaches, have been employed. Among these, green synthesis stands out for its use of environmentally friendly materials like plant extracts, bacteria, fungi, and microalgae, offering a sustainable and economically viable alternative for large-scale nanoparticle production [7].

The papaya (*Carica papaya*), a member of the *Caricaceae* family, is renowned worldwide for its nutritional and medicinal qualities. Various parts of the papaya plant have been utilised for medicinal purposes since ancient times. This article reviews the potential of papaya leaf extracts in antiviral, antidiabetic, anticancer, and anti-inflammatory applications [8]. The study conduct Ph-Fe3O4-NPs synthesised and characterization to explores the use of *Carica papaya* as a reducing agent in the green synthesis of magnetite nanoparticles, investigating the effects of temperature and reducing agent concentration on solution absorbance.

2. Materials and Method

2.1 Materials

The materials used are sodium hydroxide (NaOH) and Ferric chloride hexahydrate (FeCl3.6H2O, AR), and deionised water. The method conducted in this study as per Yew et al., [6] and Parajuli et al., [9] with some modifications, see Fig. 1.

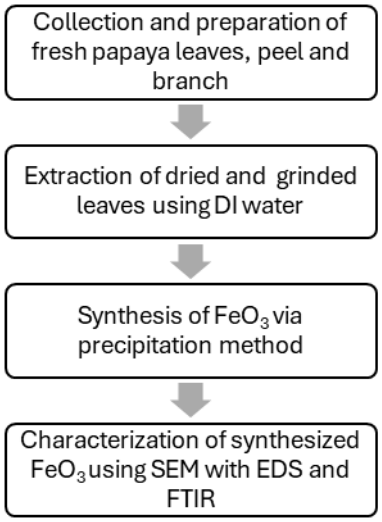

Fig. 1 *Flowchart of experiment*

2.2 Preparation of *Carica Papaya* Extract

Fig. 1 illustrates the preparation of *Carica papaya* extract from leaves, branches, and peel. Each part was cut into smaller pieces, washed with deionised water, and dried at 60°C for 24 hours. After the drying process, each part was blended into dust form for the extraction process. In this stage, each part was weighed for 10 g using an analytical balance and heated with 100 ml deionised water at 70°C for 3 hours, with regular checks using a thermometer until the colour changed [8]. The extract was then filtered through a strainer, followed by a second filter paper filtration to remove small fibres. The extracts were labelled as CP-EL (leaves), CP-EP (peel), and CP-EB (branches) and stored in the refrigerator at 4°C.

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2.3 Synthesis of Magnetite Nanoparticles

This study synthesised magnetite nanoparticles (Fe_3O_4) following Table 1 in which 2.7 g $FeCl_3.6H_2O$ was dissolved in 100 mL of deionised water in a 100 mL volumetric flask and was pipette into 250 ml beaker-filled 100 ml extract for each part. This process takes 30 minutes with stirring using a magnetic stirrer and under atmospheric pressure [6]. After 30 minutes, continue stirring the mixture for 24 hours, and the yellowish colour changed to reddish-brown. After 24 hours, sodium hydroxide aqueous solution was added to the mixture to allow the magnetite precipitations to be uniformly. Adding sodium hydroxide would turn the solution into a reddish-brown mixture and subsequently into black suspended particles. The mixture was allowed to cool down to room temperature. The magnetite nanoparticles were obtained by decantation, dilution with sterile distilled water, and centrifugation to remove the heavy biomaterials of *Carica papaya* extract. The magnetite nanoparticles were purified by dispersing in sterile distilled water and centrifugation three times at 1000 rpm for 10 minutes. The magnetite nanoparticles, after purification, were dried overnight at 50°C for XRD, EDS and FTIR analysis. The synthesis of magnetic nanoparticles was shown in Fig. 2.

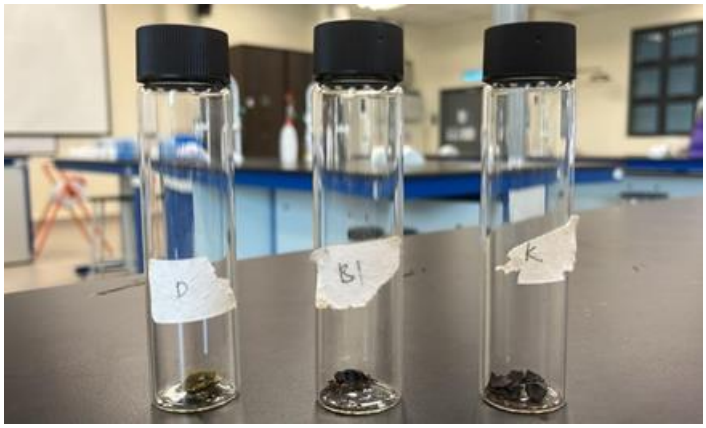


Fig.2 *Sythesis of magnetic nanoparticles*

2.4 Characterisation

The preparation of iron nanoparticles (MFeNPs) was based on the green synthesis method, where extracts of natural material are CP-EL, CP-EP, and CP-EB. The synthesised Fe_3O_4-CP and commercial Fe3O4 nanoparticles will be characterised using Scanning Electron Microscopy (SEM; JEOL-JSM 7600F, Japan) equipped with an Energy Dispersive X-ray analysis (EDS) for the surface morphology of nanoparticles. Energy-dispersive absorption Spectroscopy of derived Fe3O4 CPs confirmed the presence of elemental iron by signals ranging from 6 to 6.5 keV [1]. The sample will be secured on double-sided carbon conductive tape and sputter-coated for five minutes with a thin layer of platinum before being subjected to SEM analysis. Fourier transform infrared (FT-IR) spectroscopy was used to study the presence of the biomolecules and the functional group responsible for synthesising Fe_3O_4-NPs.

Table 1 *Formulation synthesis nanoparticle*

Formulation	Percentage of material	Composition of material
CP-EL nanoparticle	1.33 % iron (iii) chloride	2.7 g iron (iii) chloride
	49.33 % CP-EL	100 ml extract leaf
	49.33 %distilled water	100 ml distilled water
CP-EP nanoparticle	1.33 % iron (iii) chloride	2.7 g iron (iii) chloride
	49.33 % CP-EP	100 ml extract leaf
	49.33 % distilled water	100 ml distilled water
CP-EB nanoparticle	1.33 % iron (iii) chloride	2.7 g iron (iii) chloride
	49.33 % CP- EB	100 ml extract leaf
	49.33 % distilled water	100 ml distilled water

3. Result & Discussion

3.1 Fourier Transform Infrared Spectroscopy (FTIR)

The sample of magnetic nanoparticles was labelled as B for CP-EB, K for CP-EP, and D for CP-EL. Fig. 3 shows the FTIR spectrum graph illustrating the presence of various functional groups in three samples labelled B, D, and K. The x-axis represents the wavenumber in cm^{-1}, while the y-axis shows the percentage transmittance or absorbance. The graph contains colour-coded bands indicating specific functional groups: O-H stretch (hydroxyl) around 3500-3200 cm^{-1} in orange, C-H stretch (alkyl) around 3000-2850 cm^{-1} in yellow, C=O stretch (carbonyl) around 1750-1700 cm^{-1} in green, asymmetric stretch around 1650-1550 cm^{-1} in cyan, and Fe-O stretch around 550-500 cm^{-1} in pink.

All three samples exhibit the presence of these functional groups but with varying intensities and peak sharpness. Sample B shows strong peaks in the hydroxyl, carbonyl, and alkyl regions, indicating a significant presence of O-H, C=O, and C-H groups. The peaks in the asymmetric stretch region are also noticeable, and the Fe- O stretch is present, suggesting iron-oxide bonds. Sample D is similar to B but slightly lower intensity in the C-H stretch region. It shows comparable peaks in the hydroxyl and carbonyl regions, with some variation in the asymmetric stretch, indicating differences in the asymmetric bonds. The Fe-O stretch is also present.

On the other hand, Sample K shows lower intensity in the hydroxyl region compared to B and D but similar intensity in the C-H and carbonyl regions. The asymmetric stretch region displays slight differences in intensity, suggesting variations in the structural environment. The Fe-O stretch is detectable in all samples, indicating the presence of iron-oxide bonds.

In summary, while all three samples contain the same functional groups (O-H, C-H, C=O, asymmetric, and Fe-O stretches), the peak intensity and sharpness variations suggest differences in these functional groups' concentration and structural environment. Sample B generally exhibits the strongest peaks, indicating a higher concentration of these groups than samples D and K.

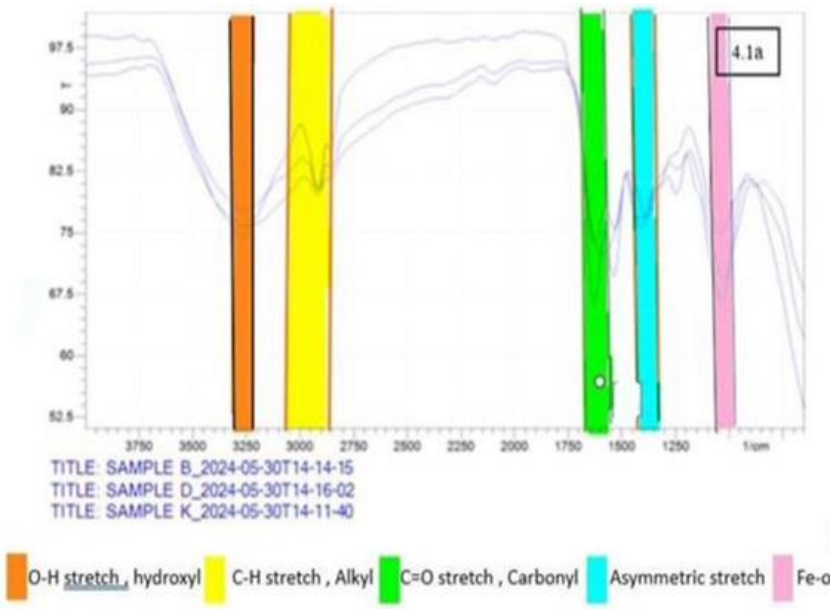


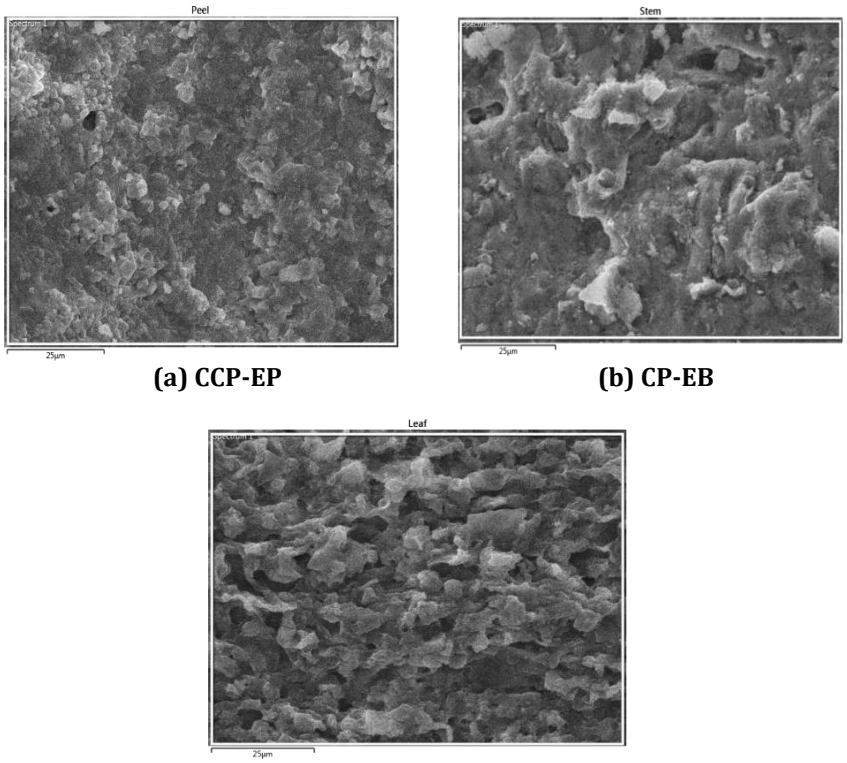
Fig. 3 *(FTIR) Result of sample EL, EP and EB*

3.2 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) analysis of magnetic Fe_3O_4 nanoparticles (MFeNPs) synthesised using *Carica papaya* CP-EL, CP-EP, and CP-EB extracts reveals critical insights into their morphological characteristics. The SEM images indicate a relatively uniform distribution, with an average particle size of approximately 25µm, microns reflecting a controlled synthesis process. In Fig. 4 CP-EP, the nanoparticles synthesised using the CP- EP extract exhibit a predominantly spherical morphology with smooth surfaces, although occasional roughness or irregularities are observed due to the organic capping agents from the papaya peel. Fig. 4 CP-EB shows the nanoparticles synthesised using the CP-EB extract, where a similar spherical shape and size uniformity are present, with some degree of agglomeration likely due to the magnetic nature of MFeNPs. The organic molecules from the branch extract help mitigate extensive clustering, resulting in smaller, manageable clusters rather than large aggregates. Fig. 4 CP-EL presents the nanoparticles synthesised using the CP-EL extract, displaying a thin

organic layer on the surfaces of the nanoparticles, indicative of effective functionalization that enhances their stability, dispersibility, and biocompatibility.

Further morphological features such as porosity and surface indentations are observed in all three samples, which could be advantageous for applications like catalysis or drug delivery due to increased surface area. The SEM images also highlight the consistency of these morphological features across nanoparticles synthesized from different parts of the *Carica papaya* plant, suggesting a robust synthesis method. The detailed morphology analysis provided by SEM confirms the successful synthesis of MFeNPs with desirable traits. The uniformity in size, spherical shape, effective functionalization, and observed porosity suggest a reliable and efficient synthesis method using *Carica papaya* extracts. These characteristics make the nanoparticles suitable for various applications requiring stable, biocompatible, and high surface area materials, thereby enhancing their potential utility in fields such as biomedicine and environmental remediation.



(a) CCP-EP **(b) CP-EB**

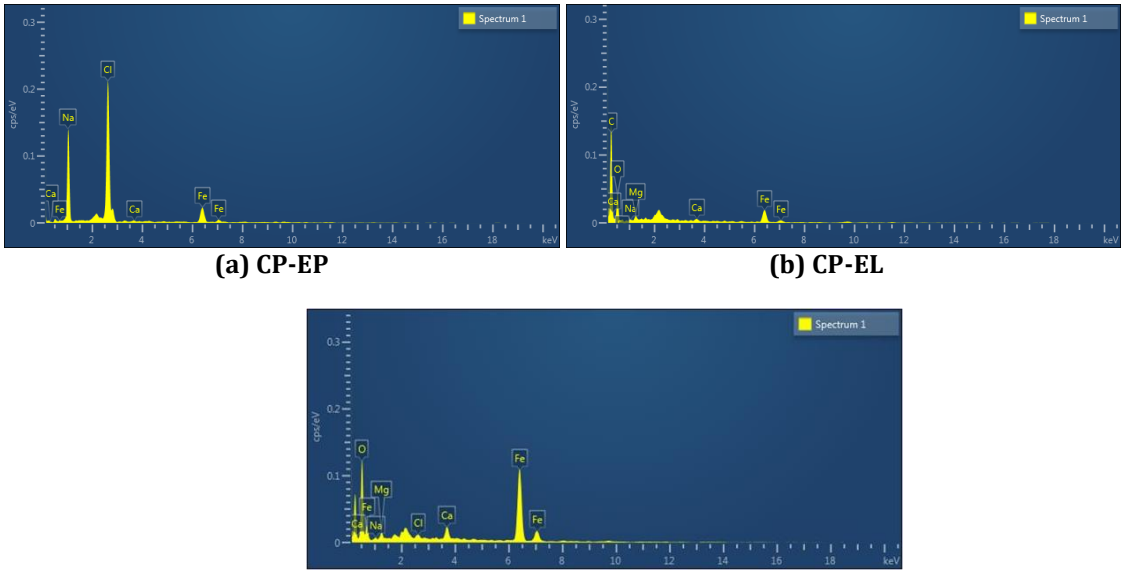
(c) CP-EL

Fig. 4 *SEM morphology figure CP-EP, CP-EL And CP-EB*

3.3 Energy Dispersive X-ray Spectroscopy (EDS)

The Energy Dispersive X-ray Spectroscopy (EDS) analysis provides valuable insights into the elemental composition of magnetic Fe_3O_4 nanoparticles (MFeNPs) synthesised using *Carica papaya* extracts. EDS detects characteristic X-rays emitted from the sample when it is bombarded with electrons, allowing for identifying and quantifying elements present in the nanoparticles. In the case of MFeNPs synthesised with CP-EL, CP-EP, and CP-EB extracts, EDS confirms the presence of iron (Fe) and oxygen (O), consistent with Fe_3O_4 composition. Carbon (C) peaks are also observed, originating from the organic capping agents derived from the papaya extracts, which contribute to stabilising and functionalisation the nanoparticles.

Based on Fig. 5, CP-EB (branch extract) exhibits the most promising results from the EDS analysis. CP-EB nanoparticles show higher iron and oxygen concentrations than CP-EL and CP-EP nanoparticles. This indicates a more efficient reduction and stabilisation process facilitated by the organic molecules present in the branch extract. The higher iron and oxygen content in CP-EB nanoparticles suggests enhanced purity and composition uniformity, crucial for applications requiring precise control over nanoparticle characteristics. Furthermore, the EDS results underscore CP-EB's effectiveness in producing MFeNPs with desirable elemental ratios, highlighting its potential for applications in biomedicine, environmental remediation, and other fields where purity and composition play pivotal roles.



(a) CP-EP **(b) CP-EL**

(c) CP-EB

Fig. 5 *EDS element result (a) peel (b) leaf (c) branch*

4. Conclusion

The magnetic nanoparticles that use iron (iii) chloride and the process of green synthesis with the extract of papaya leaves, peels, and branches have succeeded. This is because results from FTIR, SEM, and EDS tests stated that the ions of Fe, which are iron, exist in the nanoparticles tested. This shows that the nanoparticles tested indeed contain magnetic force because of the Fe ions in the nanoparticles. This experiment shows that in producing magnetic nanoparticles, the lowest toxicity rates were observed because different parts of *Carica papaya* played the role of reducing agents to reduce toxicity. It also shows that in the results, each part of *Carica papaya* stated different results, which classify the characteristics of one another. Based on the result, CP-EB (branches extract) produced better nanoparticles because of the existing Fe ions, proven in the EDS test.

Acknowledgement

The authors would like to thank the Department of Science and Mathematics, Universiti Tun Hussein Onn Malaysia for its support.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** Muhammad Izwan Hafifi Mohamad Rojie, Nurul Asyikin Mohamad Hakimi, Muhammad Hilman Daniel Abd Ghani, Mohd Khairul Nizam Mohd Zuhan*;* ***data collection:*** Muhammad Izwan Hafifi Mohamad Rojie, Nurul Asyikin Mohamad Hakimi, Muhammad Hilman Daniel Abd Ghani*;* ***analysis and interpretation of results:*** Muhammad Izwan Hafifi Mohamad Rojie, Nurul Asyikin Mohamad Hakimi, Muhammad Hilman Daniel Abd Ghani, Mohd Khairul Nizam Mohd Zuhan*;* ***draft manuscript preparation:*** Muhammad Izwan Hafifi Mohamad Rojie, Nurul Asyikin Mohamad Hakimi, Muhammad Hilman Daniel Abd Ghani, Mohd Khairul Nizam Mohd Zuhan*. All authors reviewed the results and approved the final version of the manuscript.*

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