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Green Synthesis and Characterization of Iron Oxide Nanoparticles using *Prosopis Africana* Leaf Extract

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Abstract: *Green synthesis of metal oxide nanoparticles has several advantages that include environmental friendliness. Arising on the usefulness of iron oxide nanoparticles (FeNPs) in several research and industrial quarters, we are reporting our current research outputs on the green synthesis of iron oxide nanoparticles (Fe_3O_4-NPs) from Prosopis Africana leaf extract. The synthesized Fe_3O_4-NPs were characterized by UV-vis spectroscopy, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), Fourier transforms infrared spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET)/Barrett-Joyner-Halenda (BJH) analysis. The FTIR spectroscopy confirms the presence of phytochemicals in the extract for the reduction of metal ions to nanoparticles. SEM micrograph shows that the synthesized nanoparticles are spherical with sizes ranging from 30 nm – 100 nm. BET/ BJH analysis shows that the synthesized nanoparticles are microporous with a specific surface area (46.6 m^2/g), pore volume (0.022 cm^3/g) and pore size (1.79 nm). The XRD pattern revealed the amorphous nature of the synthesized nanoparticles and the UV-vis spectrum showed a characteristic peak at 400 nm for Fe_3O_4 nanoparticles.*

Keywords: *Green synthesis, iron oxide nanoparticles, microporous, plant extract, amorphous*

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1.0 Introduction

Nanotechnology has widely been accepted as one of the most demanding research areas because of the unique properties of nanomaterials, defined as materials with particle sizes that range below 100 nm (Eddy *et al*., 2022a,b). Special properties have also positioned nanomaterials on the scale above other materials including particle size, porosity, thermal stability, optical, electrical and mechanical properties (Eddy *et* al., 2023).

Depalia *et al*., 2019). Nanomaterials have shown significant applications in various aspects sectors of materials application, either in fully fabricated form or as nanocomposites (Laurent *et al*., 2008).

Among the nanoparticles, metal nanoparticles have gained much importance due to their special nature, ease of synthesis and availability of raw materials. Several authors have stated that metal nanoparticles and metal oxide nanoparticles have a unique large surface-to-volume ratio, which can be adjusted to enhance materials' properties and subsequent applications (Ogoko *et al*., 2023). Some literature has also presented metal-based nanoparticles as essential materials in environmental remediation at various levels such as adsorption, photocatalytic degradation, nanofiltration, etc (Garg *et al*., 2021). The success of the various applications nanoparticles can facilitate depends on the materials' properties, for example, their large surface area can enhance adsorption(Astruc *et al*., 2005), and narrow bandgap can enhance their application as photocatalysts for the degradation of toxic organic contaminants (Eddy *et al*., 2023).

Although the availability of several synthetic routes for metal nanoparticles is widely acknowledged, the most widely applied methods include (i) chemical methods such as chemical reduction, and co-precipitation. (ii) physical methods such as ultrasound irradiation, sol-gel, hydrothermal methods, etc (Wu *et al*., 2015). However, these methods are expensive, not easily accessible and could generate some toxic materials in addition to the high levels of technology, some of them may require. Consequently, new cost-effective and eco-friendly methods which employ biological sources such as microbial or plant extract to synthesize nanoparticles have been widely recommended (Joergear *et al*., 2000). The eco-friendly method, which translates to green synthesis has proven to be a better alternative to chemical methods because the dominant reagents for the methods are harmless, especially those involving plant extracts. In the green synthesis technique, an environmentally friendly solvent, capping agent and reducing agents are fundamental materials required (Prasad *et al*., 2015). Green synthesis of nanoparticles, using plant extract has been widely reported and is known to involve the reduction roles of biomolecules (such as alkaloids, terpenoids, phenolic compounds, peptides, proteins and free amino acids) in reducing metal slats to their nanoparticles(Chauhan and Uphadhyay, 2019; Páez *et al*., 2019).

Green synthesis of iron oxide nanoparticles (FeO-NPs) using plant extracts has been reported for some combination of plant extract and metal salts. For instance, Sentil and Ramesha (2012) synthesized Fe_3O_4 nanoparticles from *Tridax Procumbens* leaf extract, and the results they obtained gave evidence that indicated that aldehyde groups in the water-soluble carbohydrates were responsible for the reduction of the metal salt to Fe_3O_4 nanoparticles. The synthesized nanoparticles were characterized and observed to show a strong antibacterial effect on *Pseudomonas aeruginosa*. Kanagasubbulakshmi and Kadirvelu (2017) synthesized iron oxide nanoparticles (FeO-NPs) using *Lagenaria siceraria* extract, the synthesized FeO-NPs were characterized using UV-vis , SEM, EDX, XRD, Zeta sizer, and FT-IR. It was observed that the synthesized FeO-NPs were within the size range of 30 nm – 60 nm. A study on the antimicrobial activity of the FeO-NPs was carried out against Gram-negative - *Escherichia coli* and Gram-positive- *Staphylococcus aureus*, an inhibition zone of 10 mm and 8 mm was observed for *Escherichia coli* and *Staphylococcus aureus* respectively. Iron nanoparticles have been widely reported as efficient agents for environmental pollution remediation (Sharma *et al*., 2018). for the application of the compound for the photocatalytic degradation of dyes and other

organic pollutants as well as adsorption removal of several grades and types of water contaminants are extensively documented in the literature (Eddy *et* al., 2022a-b, 2023; Shakhawat *et al*., 2020; Yadav *et al*., 2020). Iron nanoparticles have shown removal efficiency between 88.34% and 85.94% for total petroleum hydrocarbons (TPHs) from water and 81.90% from soil (Erika *et al*., 2018). The suitability of iron nanoparticles for remediation is a result of their non-toxicity, magnetic susceptibility and dual redox capability on reaction with water, it also possesses a large surface area and high reactivity (Bolade *et al*., 2020).

Prosopis Africana is usually found in the savannah region of West Africa. It belongs to the family of Leguminosae, and its subfamily is Mimosoideae. Phytochemical analysis of the leaves showed the presence of alkaloids, saponins, tannins, glycosides, flavonoids and anthraquinones in petroleum ether, ethyl acetate, methanol and water extracts (Elaigwu *et al*., 2018; Obode *et al*., 2021). Hence, extract from *Prosopis Africana* could reduce metal ions to nanoparticles due to the presence of these phytochemicals that can act as reducing and stabilizing agents. This study therefore aimed at green synthesis and characterization of iron oxide nanoparticles (magnetite, Fe_3O_4-NPs) using *Prosopis Africana* leaf extract.

2.0 Materials and Methods

2.1 Materials

Iron (III) nitrate nonahydrate (Fe $(NO_3)_3.9\ H_2O$) and sodium hydroxide (NaOH) were obtained from Sigma-Aldrich Chemicals. Deionized water was used throughout the synthesis. All reagents used in the study were of analytical grade. *Prosopis Africana* leaves were obtained from a garden in Area BZ of Ahmadu Bello University, Zaria; they were stored in a new clean polyethene bag and taken to the laboratory for green synthesis of the Fe_3O_4-NPs.

2.2 Preparation of plant extract

The fresh leaves collected were washed thoroughly severally with tap water to remove dust particles, followed by proper rinsing with distilled water. The leaves were air-dried for 10 days and then ground to a fine powder with a Thomas-Wiley laboratory mill (Model 4) and stored in a clean polythene bag for further use. 25 g of fine powder was boiled with 200 mL of deionized water for 5 min and the extract was then filtered using Whatman filter paper no 1. The aqueous plant extract was collected in an amber-coloured bottle and stored at 4°C for further use. (Ravichandran *et al*., 2019).

2.3 Green synthesis of Fe_3O_4 nanoparticles (Fe_3O_4-NPs)

The synthesis involved the use of iron (III) nitrate nonahydrate (Fe $(NO_3)_3.9\ H_2O$) as the precursor while freshly prepared plant extract was used as a reducing and stabilizer agent. A solution of 0.01 M Fe $(NO_3)_3.9\ H_2O$ was prepared by adding 1.01 g of solid Fe $(NO_3)_3.9\ H_2O$ in 250 mL deionized water and stirred for 30 min. The aqueous solution containing the precursor was heated in a hot plate set at 60 ^{0}C for 5 min, under constant stirring. . For the green synthesis of iron oxide nanoparticles, 5 mL of the plant extract was added dropwise to 10 mL of 0.01 M Fe $(NO_3)_3.9\ H_2O$ precursor solution with continuous stirring for a homogeneous reaction. Afterwards, 1.0 M NaOH was added till the pH became 9. A brownish-black precipitate was formed after the addition of the plant extract and this is an indication of the formation of Fe_3O_4-NPs (Kanagasubbulakshmi and Kadirvelu 2017; Kiwumulo *et al*., 2022). The formation of nanoparticles was caused by the presence of functional groups like hydroxyl, carboxyl, alkyne, and aldehyde, which acts as reducing and stabilizing agent in the plant extract (Kanagasubbulakshmi and Kadirvelu 2017; Idris *et al*., 2022). The nanoparticles formed were filtered using Whatman filter paper No. 1 and subsequently washed using deionized

water to a neutral point. The dried precipitate powder was oven dried at 70^0C for 3 h to obtain black-coloured nanoparticles and stored in a seal-tight container for further use (Spivakov *et al.*, 2020).

2.4 Characterization

For an understanding of the specific properties of the synthesized nanoparticles, characterization was further carried out by subjecting the synthesized nanoparticles to UV-Vis spectroscopy, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX**)**, X-ray diffraction (XRD), Fourier transforms infrared spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET)/ Barrett-Joyner-Halenda (BJH) analysis.

3.0 Results and Discussion

3.1: UV-Vis spectroscopy of synthesized iron oxide nanoparticle

The ultraviolet-visible analysis gives the optical characteristics of the green synthesized iron nanoparticles. UV-vis spectrum of the synthesized iron oxide nanoparticle from *Prosopis Africana* leaf extract is presented in Fig. 1. The UV–visible analysis was initiated at a wavelength of 200–800 nm to study the absorption spectrum of the green synthesized iron oxide nanoparticle using a UV-Vis spectrophotometer, Model Cary 300 (Agilent Technologies, USA). From the spectrum, the nanoparticles showed peaks at 400 nm and 633 nm, which is an indication of the formation of nanoparticles, the peak at 400 nm corresponds to a characteristic peak for Fe_3O_4 nanoparticles. The characteristic peak for Fe_3O_4 at 400 nm was also observed by Bahadur *et al*. (2017) when eco-friendly synthesis of magnetite (Fe_3O_4) nanoparticles with tunable size: dielectric, magnetic, thermal and optical was studied. Rajendran *et al*. (2015) and Lesiak *et al*. (2019) observed surface Plasmon resonance (SPR) band at 417 nm and 400-420 nm for Fe_3O_4 nanoparticles when studies were carried out on nanoparticles to determine efficacy for removal of arsenic from water, and surface characteristics as influenced by adsorbed biomolecules respectively. The peak at 663 nm could emanate from biomolecules contained in the *Prosopis Africana* leaf extract.

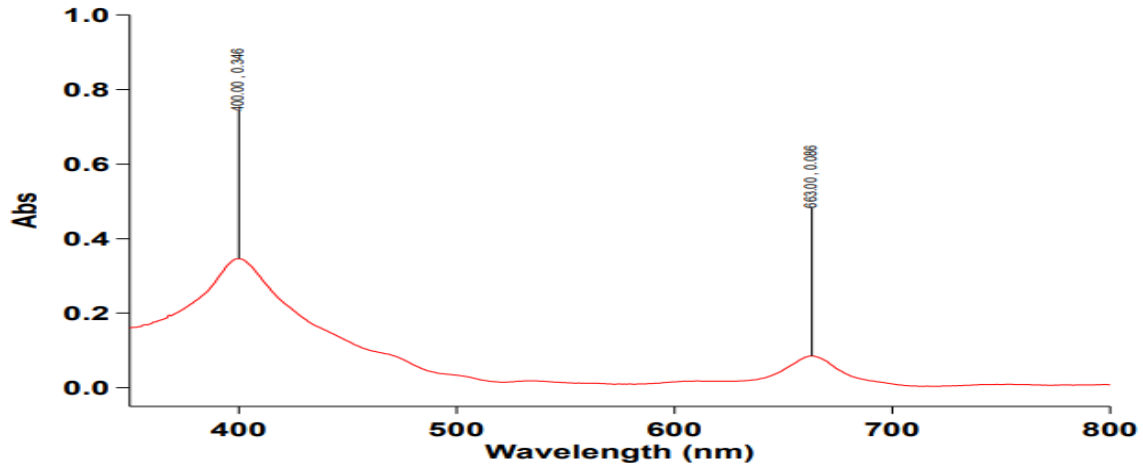

Fig. 1: UV-visible Spectroscopy of Fe_3O_4-NPs synthesized from *Prosopis Africana* leaf extract

3.2 Scanning Electron Microscopy (SEM) of the synthesized iron oxide nanoparticle

The morphological characteristic of the green synthesized Fe_3O_4-NPs was discovered by scanning electron microscopy analysis using SEM microscope that is coupled with EDX detector, Model Supra TM 35 VP (Carl Zeiss instrument, United Kingdom). The SEM micrograph at multiple magnifications as presented in Fig. 2 shows that the synthesized

nanoparticle is spherical. This conforms with what was observed by Fatimah *et al*. (2020) in the green synthesis of Fe_3O_4-NPs for photocatalytic degradation of Bromophenol blue. The spherical shape of the synthesized Fe_3O_4-NPs makes creates high potency for antibacterial activity as spherical nanoparticles penetrate into the cell wall of pathogens easily (Wiley *et al*., 2006). The SEM analysis revealed that the synthesized nanoparticle sizes ranged from 30 nm – 100 nm. Kanagasubbulakshmi & Kadirvelu (2017) obtained this same range of size when the characterization and antimicrobial activity of green synthesis Fe_3O_4-NPs from *Lagenaria Siceraria* was evaluated. This suggests that *Prosopis Africana* leaf extract and that of *Lagenaria Siceraria* contain some phytochemicals that can lead to the same Fe_3O_4-NPs characterization in terms of size.

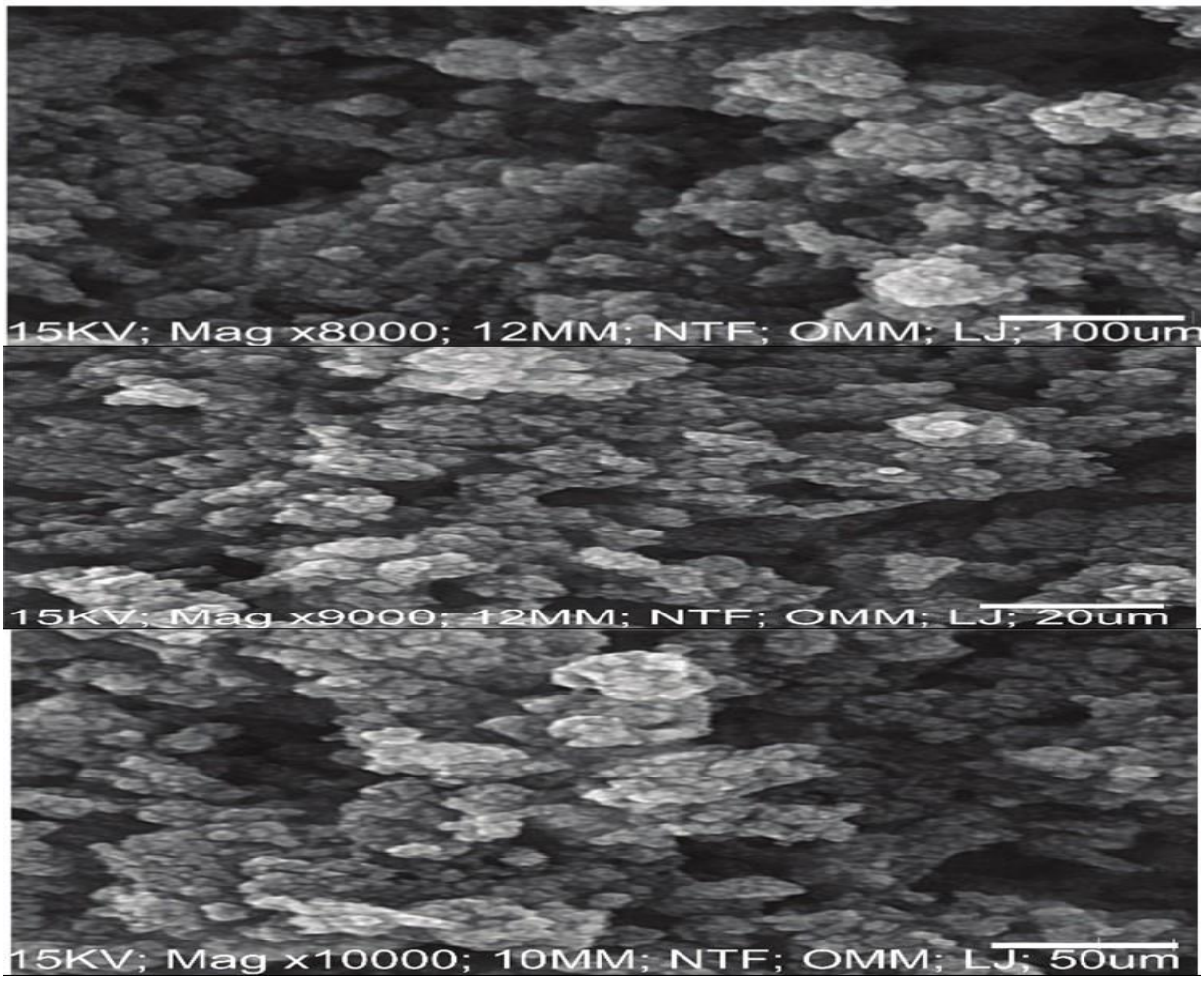

Fig. 2: SEM micrograph of the green synthesized Fe_3O_4-NPs

3.3Energy-dispersive X-Ray Spectroscopy (EDX) of the synthesized Fe_3O_4-NPs

The EDX spectra presented in Fig. 3 show the elemental composition of the green synthesized Fe_3O_4-NPs. As shown by the spectra, the elemental content is Fe (iron), O (oxygen) and C (carbon), and the atomic composition is 76.75 %, 20.79 % and 2.46 % respectively. This composition shows the high purity of the magnetite nanoparticles, the foreign element (carbon) present could be due to impurity in the nanoparticle sample (Geneti *et al*. (2022) and plant biomolecules

presence in metal ions reduction and stabilization process of the nanoparticles Kiwumulo *et al*. (2022).

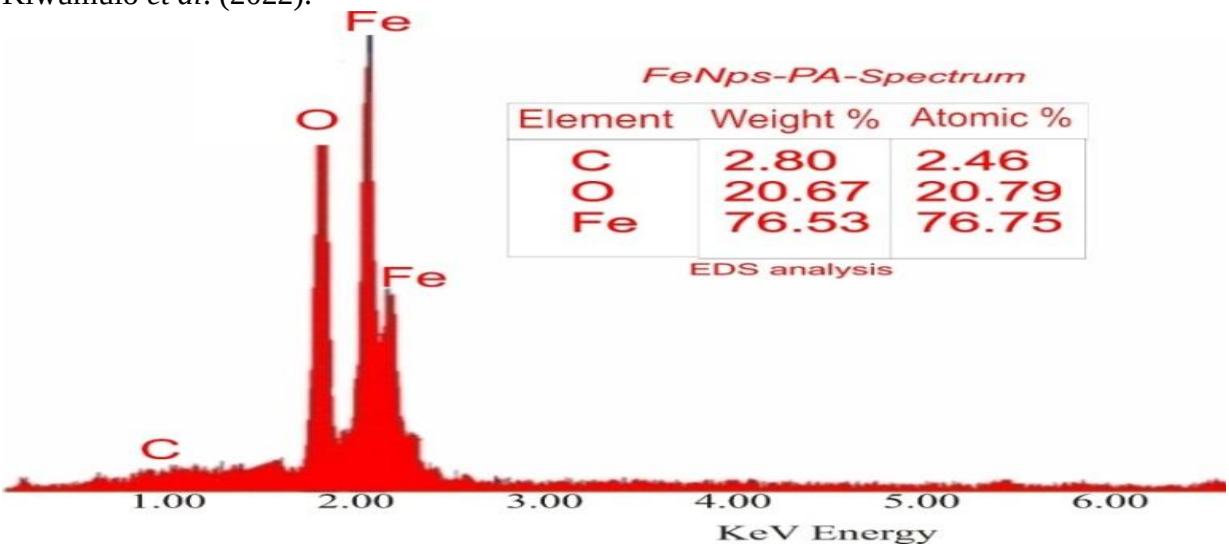

Fig. 3: EDX of the green synthesized Fe_3O_4-NPs

3.4 X-Ray diffraction (XRD) analysis of the green synthesized Fe_3O_4-NPs

X-Ray diffraction (XRD) analysis was carried out using X-Ray Diffractometer, Model Miniflex 600 (Rigaku Corporation, Japan) to determine the crystallinity of the synthesized nanoparticles. The XRD pattern of the synthesized Fe_3O_4-NPs as presented in Fig. 4 shows no characteristic diffraction peaks, but a broad spectrum. This is because, the synthesized nanoparticles are amorphous, and no crystalline phase was formed. The amorphous structure is attributed to little impurity that could disrupt the lattice. It can also be deduced that the percentage of the amorphous particles formed, supersedes the crystalline particles contained in the nanoparticles (Bayat *et al*., 2021). This amorphous structure can improve the solubility and bioavailability of the synthesized Fe_3O_4-NPs for medical applications (Jog and Burgess, 2017). The syntheses of iron nanoparticles with amorphous structures have been reported. For instance, Ebrahiminezhad *et al*., (2017) in the study 'green synthesis and characterization of zerovalent iron nanoparticles from *Urtica dioica* leaf extract, obtained iron nanoparticles with an amorphous structure, Yadav *et al*. (2020) has also synthesized iron oxide nanoparticles with an amorphous structure, though the sonochemical method.

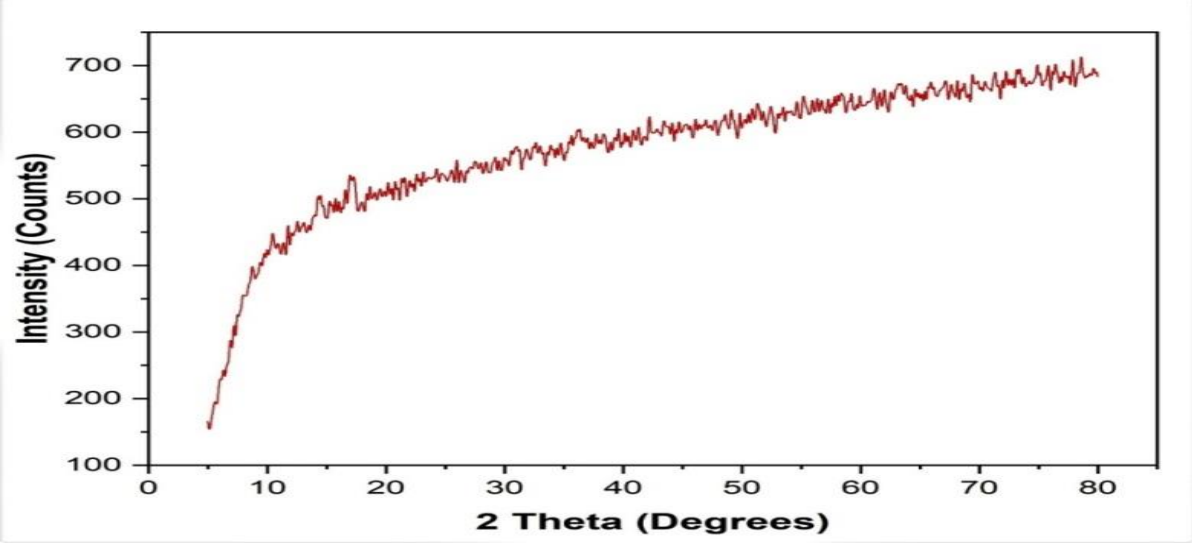

Fig. 4: XRD pattern of the green synthesized Fe_3O_4-NPs

3.5 Fourier Transform Infrared (FTIR) Spectroscopy of the green synthesized iron oxide nanoparticles

Fourier transform infrared spectroscopy (FT-IR) analysis was carried out using FT-IR spectrophotometer, Model Carry 630 (Agilent Technologies, USA). The synthesized nanoparticle (10 mg) was measured with a resolution of 4 cm^{-1} and a scan range from 400 to 4000 cm^{-1} (Devi *et al*., 2019). The FT-IR spectra show the functional groups in the plant extract and the interaction between the biomolecules in the plant extract and the metal ions. The FT-IR spectrum of the synthesized iron oxide nanoparticle is presented in Fig. 5. The band at 1560 cm^{-1} corresponds to N-H bending of amine, and the band at 1066 cm^{-1} is due to characteristic vibration stretching of C-O and C-OH from the phytochemicals present in the plant extract. The absorption bands at 420.92 cm^{-1} - 402 cm^{-1} are due to Fe-O stretching which are in close agreement with the report by Hassan *et al*. (2020) at 428.27 cm^{-1} in the green synthesis of bentonite-supported iron nanoparticles as a heterogeneous Fenton-like catalyst. Furthermore, Parajuli *et al*. (2020) observed Fe-O stretching band at 404 cm^{-1} when magnetite nanoparticles (Fe_3O_4-NPs) were synthesized from *Azadirachta indica* leaves extract.

3.6 Surface Area and Pore Analysis of the Green Synthesized Iron Oxide Nanoparticles

The surface area and pore characteristics of the synthesized Fe_3O_4-NPs were determined by applying Brunauer–Emmett–Teller (BET) analysis for surface area characteristics and Barrett-Joyner-Halenda (BJH) analysis for pore characteristics. Analysis for specific surface area, pore volume and pore size of the nanoparticles was carried out using Automated Gas Sorption Analyzer, which is a combined BET and BJH analyzer, Model Nova Station A (Quatachrome Instrument, USA). From the analysis as presented in Table 1, the specific surface area of the synthesized nanoparticles is 46.6 m^2/g, the pore volume is given to be 0.022 cm^3/g, and the pore size is 1.79 nm. Hence, the iron oxide nanoparticles synthesized are microporous. In the green synthesis and characterization of mesoporous magnetite nanoparticles from *Peltophrorum pterocarpus* extract, Dash *et al*. (2019) obtained a specific surface area of 47.07 m^2/g for the nanoparticles, which is similar to the

specific surface area of 46.6 m^2/g obtained in this work. Furthermore, Mishra & Ramaprabhu (2011) obtained a similar pore size in the range of 1.7-2.5 nm when a study on the adsorption of carbon dioxide by nano magnetite was conducted. The microporous nature of the synthesized Fe_3O_4-NPs can enhance their antibacterial activity and other medical applications due to their well-defined porous structures for drug delivery and efficacy (Wan *et al*., 2020)

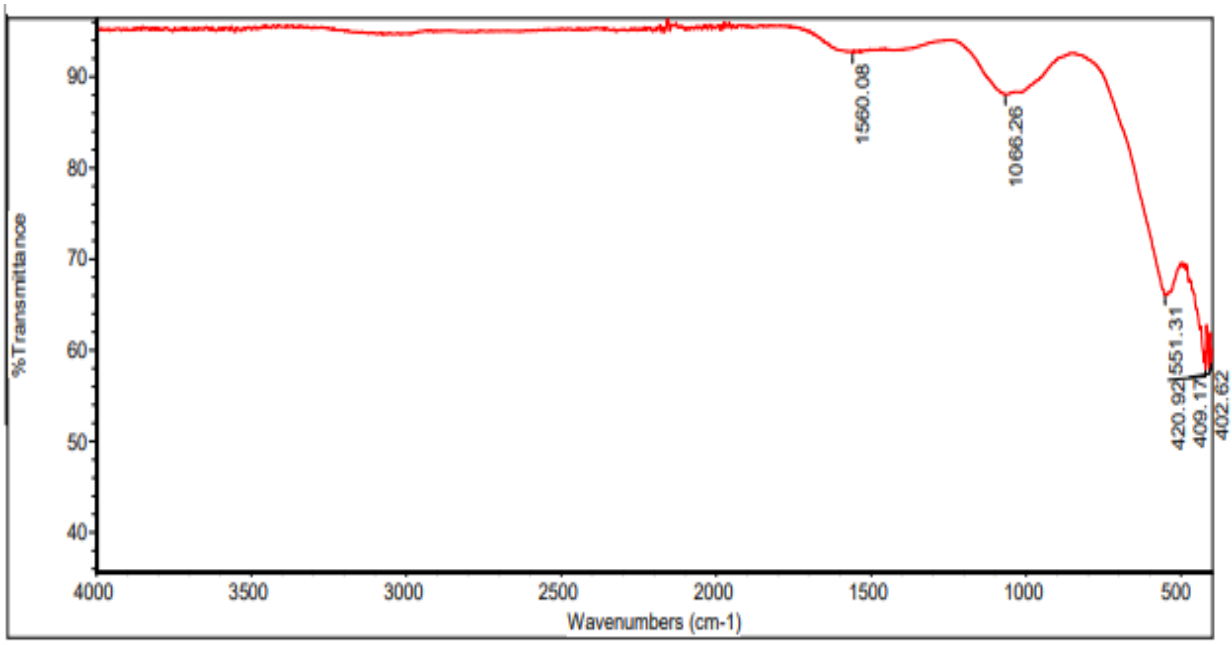

Fig. 5: FTIR spectrum of the synthesized iron oxide nanoparticles

Table 1: Surface Area and Pore distribution of the Green Synthesized Fe_3O_4-NPs

Parameters	Analyzed Value
specific surface area (m^2/g) BET	46.6
pore volume (cm^3/g) BJH	0.022
pore size (nm) BJH	1.79

4.0 Conclusion

Green synthesis of iron oxide nanoparticles was carried out using *Prosopis Africana* leaf extract, phytochemicals in the extract as responsible for reducing the iron (III) nitrate nonahydrate to iron oxide nanoparticles (Fe_3O_4 NPs). The nanoparticles were characterized by UV-vis spectroscopy, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET)/Barrett-Joyner-Halenda (BJH) analysis. SEM micrograph showed that the synthesized nanoparticles are spherical with sizes ranging from 30 nm – 100 nm. FTIR confirms the presence of phytochemicals for the reduction of metal ions to nanoparticles and the presence of Fe-O bond. BET/BJH analysis showed that the nanoparticles are microporous with a specific surface area (46.6 m^2/g), pore volume (0.022 cm^3/g) and pore size (1.79 nm). XRD pattern revealed the amorphous nature of the synthesized nanoparticles and the UV-vis spectrum showed a characteristic peak at 400 nm for Fe_3O_4 nanoparticles. EDX reveals high purity of the nanoparticles with an atomic composition of 76.75 %, 20.79 % and 2.46 % for Fe, O and C respectively.

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