THE GREEN SYNTHESIS OF IRON NANOPARTICLES FROM AN AQUEOUS EXTRACT OF NEEM LEAVES (*Azadirachta indica*)

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INTRODUCTION

Green synthesis is a function that replaces the reducing agent with a natural extract from plant materials such as leaves, flowers, and seeds (Eslami et al., 2018). The green synthesis of nanoparticles does not require high pressure, energy and temperature conditions and toxic chemicals. This technology is the most appropriate for the manufacturing of metal nanoparticles from plant component extracts (Tyagi et al., 2021; Gholami et al., 2018). For this investigation, FeNPs were employed as iron metal, which is a non-toxic, easily produced, environmentally friendly and less expensive metal nanoparticle. FeNPs are employed in a variety of applications including medicine, wastewater treatment, coatings and textile applications (Saranya, 2017; Ksv et al., 2017). However, the FeNPs were created using a green synthesis method, by means of an aqueous extract of neem leaves as the reducing agent. The neem tree is native to South Asian countries, but its cultivation has already spread to countries on other continents with tropical climates. It is used in traditional medicine treatments. This study consults and discusses the application of the neem tree in agriculture, medicine and the environment (Roshan & Verma, 2016; Hashmat et al., 2021). Furthermore, this research investigates the reduction and stabilization effect of the aqueous extract of neem leaves using the synthesis of iron nanoparticles (FeNP) and the characterization of synthesized FeNPs using Scanning Electron Microscope (SEM) analysis, Energy Dispersive X-Ray (EDX) analysis, X-ray diffraction (XRD) analysis and UV-Visible Spectroscopy (UV-Vis) analysis.

METHODOLOGY

Collection of the plant and chemical materials

Fresh neem leaves were collected from a village in Sri Lanka. Ferric chloride hexahydrate ($FeCl_3.6H_2O$) was used as the source of iron (Fe) and was obtained from a local chemical supplier. The dialysis membrane tubing (Molecular weight cut off 12,000-14,000 Dalton) used in this work was also purchased from a local chemical supplier. The distilled water was prepared using the Distilled Water Dispenser in the laboratory. In this study, all chemicals used were of analytical grade and utilized without any additional purification.

Preparation of the extract of dry neem leaves

The collected fresh leaves of neem were washed several times with water to remove dust and other particulate matter. The clean leaves were shade dried for a few days and finely crushed. The crushed leaves from 10 g were mixed with 100 ml distilled water and the mixture (MLR 1:10) was heated at 60-70 °C for 60 mins. After that, the mixture was cooled to room temperature and filtered. For further studies, the extract was kept at 8 °C temperature.

Synthesis process of Fe-Nanoparticles

The extract of neem leaves was mixed with a 0.1M ferric chloride ($FeCl_3.6H_2O$) solution in a volume ratio of 1:2 at room temperature. The resultant mixture was stirred using a magnetic stirrer at 300 rpm for 60 minutes and the formation of an intense black-coloured solution confirmed the synthesis of iron nanoparticles. The mixture was centrifuged at 5000 rpm for

20 minutes and separated for the FeNPs to precipitate (this process was done by two or three times). The supernatant was discarded, and the dialysis membrane was used to dialyze precipitate 2-3 times with distilled water. Eventually, the FeNPs were dried at 60 °C in a vacuum oven and stored in a seal-tight container for further use (Pan et al., 2020).

Characterization of synthesized Iron Nanoparticle

SEM characterization

The surface morphology of the synthesized FeNPs was analyzed using a Hitachi SU6600 Scanning Electron Microscope with an Energy Dispersive X-Ray (EDX) analyser. The SEM was operated at 10 kV with a magnification of × 40,000. For the SEM analysis, the purified synthesized FeNPs were located on the SEM holder and coated on using gold sputters. The Elemental composition of the synthesized FeNPs was checked using the EDX analyser.

XRD characterization

An X-ray diffractometer was used to examine the crystalline metallic FeNPs. The X-ray diffraction spectrum (XRD) was recorded by the Bruker D8 Focus X-Ray Diffraction Spectrometer at the scanning rate of 2°/min from 2θ range of 5°-60° with Cu Kα radiation (λ = 1.5418 A°) utilizing 40 kV and 30 mA for voltage and current.

UV-Visible characterization

The optical characteristics of the produced iron nanoparticles were investigated using a UV-Visible absorption spectroscopy. The absorbance spectrum was evaluated in the wavelength range of 200 to 800 nm using a Thermo scientific genesis 10S series type UV-Visible spectrophotometer.

RESULTS AND DISCUSSION

Visual inspection of the synthesized iron nanoparticles

Figure 1 shows the synthesized nanoparticle solution using the aqueous extract of neem leaves as the reducing agent. After the vacuum dry process, the concentrated iron nanoparticles in powder form is shown in Figure 2.

Figure 1. Synthesized iron nanoparticles solution

Figure 2. Synthesized iron nanoparticles in powder

SEM analysis of synthesized Iron Nanoparticles

The SEM images of synthesized iron nanoparticles were provided with a magnification of 1 µm and 200 nm (Figure 3). It indicated that nanoparticles formed are agglomerated due to their adhesive nature of a morphology of spherical shapes with an appearance of a particle size ranging between 20 to 40 nm. The EDX analysis determined the elemental composition of FeNPs that were synthesized using the aqueous extract of neem leaves as a reducing agent. The EDX diagram is shown in Figure 4. The EDX qualification gives atomic percentages of 9.82% of iron, 44.77% of carbon, 41.71% of oxygen, 0.68% of phosphorous, 0.97% of sulphur, 1.02% of potassium and 1.03% of calcium.

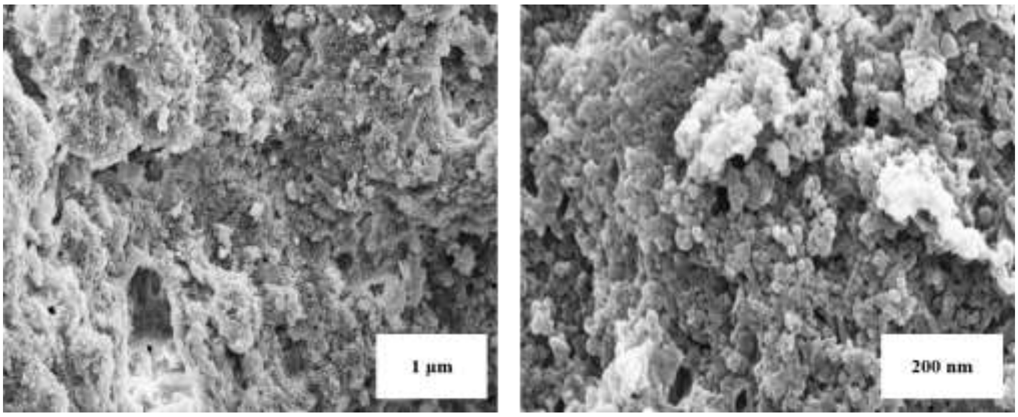

Figure 3. SEM image of synthesized FeNPs at 1 µm and 200 nm magnification

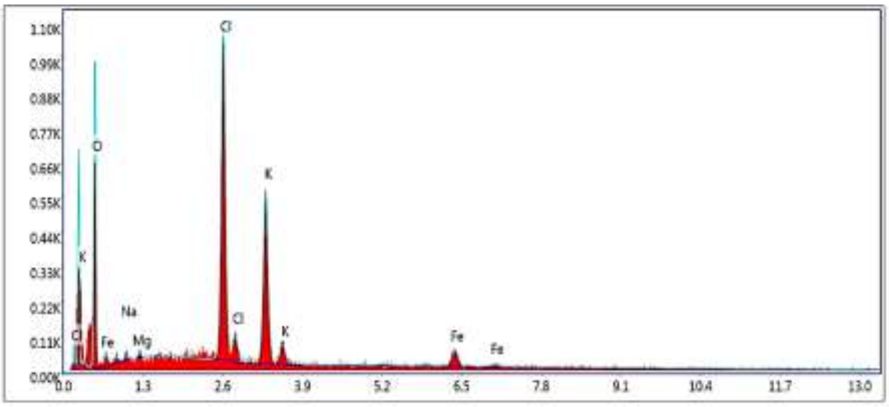

Figure 4. EDX analysis of synthesized iron nanoparticles

XRD analysis of synthesized Iron Nanoparticles

The XRD spectrum of synthesized FeNPs which were extracted using the aqueous extract of neem leaves is depicted in Figure 5. The XRD spectra show that there are significant peaks recorded at 24.16°, 33.15°, 39.27°, and 40.83°, corresponding to the phase planes of 012, 104, 006, and 113, respectively. These values have established the characteristics of the crystalline metallic iron phase.

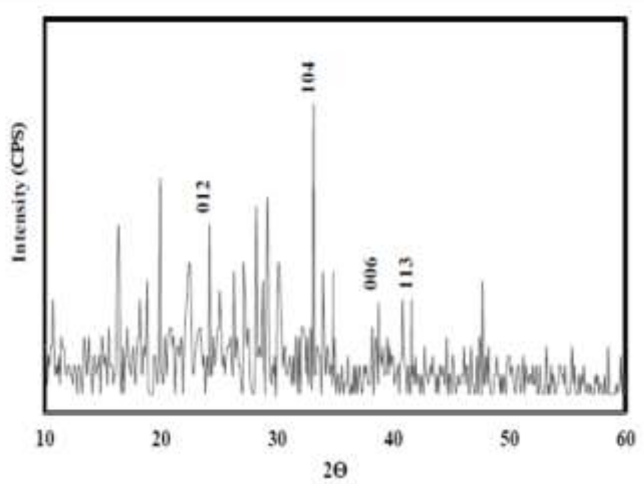

Figure 5. XRD spectrum of synthesized FeNPs

UV–Visible absorption analysis of synthesized Iron Nanoparticles

The UV-Visible spectra of the aqueous extract of neem leaves from the green synthesis of FeNPs in aqueous solutions were measured. The UV-Visible spectral analysis was done within the range of 200-800 nm and absorption peaks were observed in the 250–295 nm region due to the excitation of surface plasmon vibrations in the iron nanoparticles. The maximum peak was observed at 272 nm, which is identical to the characteristics of the UV-Visible spectrum of iron nanoparticles which was recorded (Pattanayak and Nayak, 2013). The room temperature absorption spectrum of the FeNPs nanoparticles is shown in Figure 6.

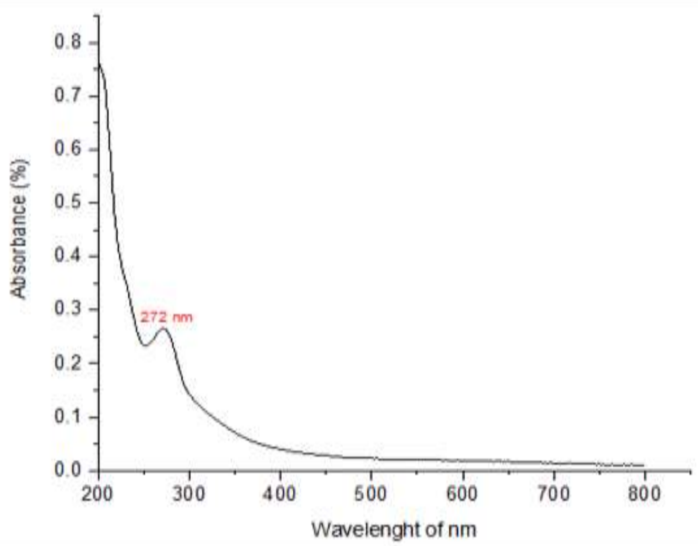

Figure 6. UV-Visible spectrum of synthesized FeNPs

CONCLUSIONS

An aqueous extract of neem leaves was utilized as a reducing and stabilizing agent for the synthesis of FeNPs. To validate the synthesis of FeNPs, many characterization approaches such as SEM, EDX, XRD and UV-Visible analyses were used. The SEM imaging reveals that FeNPs tend to aggregate into spherical shapes with the appearance of a particle size ranging between 20-40 nm. The characteristic crystalline metallic iron phase of synthesized FeNPs were identified using the XRD spectrum. Using the UV-Visible spectral analysis, absorption peaks were observed in the 250-295 nm region while the maximum peak was observed at 272 nm, which is identical to the characteristics of the UV-Visible spectrum of synthesized FeNPs. These results prove that iron nanoparticles can be competently synthesized using an aqueous extract of neem leaves as the reducing agent. This is a step toward a more environmentally friendly future in which industrial metal nanoparticle production can be done without the use of harmful reducing and capping chemicals.

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