

# Eco-Friendly Biosynthesis of Recyclable Silver Nanocatalyst and Iron Nanocatalyst using *Azadirachta indica*

Arzoo Siddiqui<sup>1\*</sup>, Nikhat Fatima<sup>2</sup>, Amit Yadav<sup>2</sup>, Amulya Sinha<sup>2</sup>, Vaibhav Tripathi<sup>2</sup>, Girish Singh<sup>2</sup>, Anmol Kumar<sup>2</sup>, Vivek Bhaduria<sup>\*</sup>

<sup>1,2,\*</sup> Green Laboratory, Department of Chemistry, Ewing Christian College, University of Allahabad, Prayagraj, Uttar Pradesh, India-211003.

Email: [arzoosiddiqui56@gmail.com](mailto:arzoosiddiqui56@gmail.com)

Email: [nikhatfatima162@gmail.com](mailto:nikhatfatima162@gmail.com)

Email: [yadavamitjoy5998@gmail.com](mailto:yadavamitjoy5998@gmail.com)

Email: [amulya.sinha28@gmail.com](mailto:amulya.sinha28@gmail.com)

Email: [vaibhavtripathi2798@gmail.com](mailto:vaibhavtripathi2798@gmail.com)

Email: [singhgirish036@gmail.com](mailto:singhgirish036@gmail.com)

Email: [anmolkumar10021997@gmail.com](mailto:anmolkumar10021997@gmail.com)

Email: [bvivek17@gmail.com](mailto:bvivek17@gmail.com)

## <sup>1\*</sup>Corresponding Author

**Arzoo Siddiqui<sup>1\*</sup>**

Green Laboratory, Department of Chemistry, Ewing Christian College, University of Allahabad, Prayagraj, Uttar Pradesh, India-211003.

Email: [arzoosiddiqui56@gmail.com](mailto:arzoosiddiqui56@gmail.com)

Mobile NO: 8299602185

**A B S T R A C T:** The environmentally friendly and sustainable method of producing silver nanoparticles (AgNPs) through the green synthesis of plant extracts has drawn a lot of interest. In this study, neem leaf extract (*Azadirachta indica*) was used as a capping and reducing agent in the synthesis of AgNPs. The color shift from greenish brown to dark brown verified the formation of AgNPs. X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and UV-Vis spectroscopy were used to analyze the biosynthesized AgNPs. AgNP production was indicated by a distinctive surface plasmon resonance peak in the UV-Vis spectra. Functional groups including hydroxyl, methyl, carboxylate, and carbonyl groups were detected by FTIR analysis, indicating the role of flavonoids and terpenoids in the stability and reduction of AgNPs. The XRD study verified that the produced AgNPs had a face-centered cubic structure and were crystalline. *A. indica* leaf extract is used in the green synthesis of AgNPs, which provides a quick, economical, and ecologically safe method for creating nanoparticles with a wide range of uses in industries such as environmental remediation, biomedicine, and catalysis. In the current study, biosynthesized iron oxide nanoparticles (Fe<sub>2</sub>O<sub>3</sub>-NPs) were successfully manufactured using a non-toxic leaf extract of

neem (*Azadirachta indica*) as a stabilizing and reducing agent. The effectiveness of *Azadirachta indica* extract as a reducing and stabilizing agent was demonstrated by the quick reduction process. Iron oxide nanoparticles with a cubic crystal structure and no impurities were visible in the XRD pattern. The interactions between *Azadirachta indica* and iron oxide nanoparticles were demonstrated by FTIR.

**Keywords:** Silver nanoparticles; Green synthesis; *A. indica*; Iron nano-catalyst.

## INTRODUCTION

Plant extracts are a promising option for use as a green reducing agent for the manufacture of metal nanoparticles and bioapplications since they are abundant in many compounds, including polyphenols, flavonoids, fatty acids, etc. Depending on the inherent qualities of plant extract, green produced silver nanoparticles (AgNPs) typically have significant biological qualities like antibacterial and antioxidant capabilities in addition to good catalytic properties because of the high conductivity of silver at the nanoscale [1–3]. The size of AgNPs has a significant impact on the catalytic and antibacterial qualities of biosynthesized AgNPs; smaller AgNPs perform better than larger AgNPs [4–8]. There are hundreds of papers on the green production of metal nanoparticles utilizing different types of plant extracts, according to a basic literature review.

The resulting nanoparticles feature both plasmonic and bio-related properties of their plant extract source, including antibacterial and antioxidant qualities, because every plant (and plant portion) has different properties [9,10–13]. Thus, research into the possibility of novel plant extracts as reducing agents for the manufacture of metal nanoparticles may lead to the discovery of novel nanomaterials with improved or novel features.

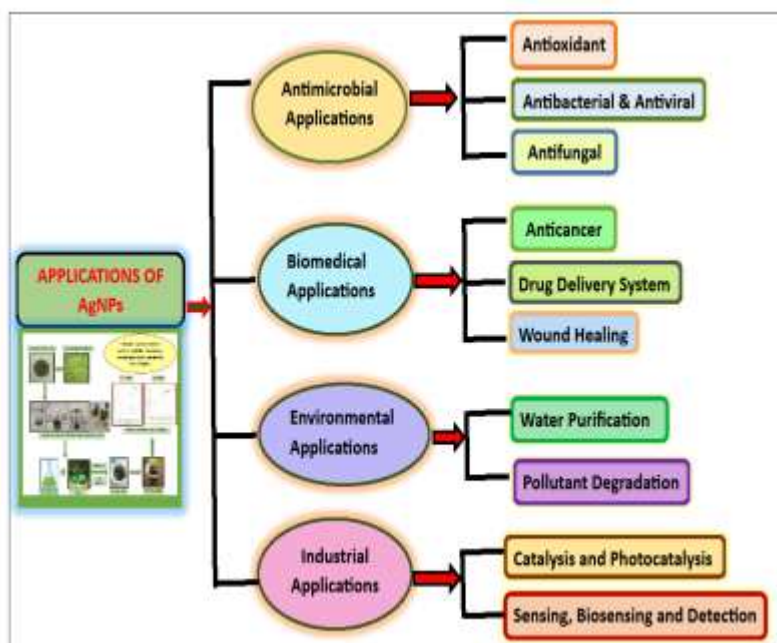
AgNPs are especially intriguing in the context of sustainable science, especially for catalysis, given their characteristics [14–21]. Numerous investigations on the effectiveness of CuInanoparticles as a coating on silicate, aluminum oxide, titanium dioxide, fiberglass, ceramics, and activated carbon have been conducted during the past ten years [22–30].

Thus, AgNP catalysis research and the creation of new, green, cost-effective, and efficient pathways for AgNP-catalyzed chemical processes remain significant challenges in a very active field.

This study attempted to create silver nanoparticles by using *A. indica* leaf extracts as a capping and reducing agent. The produced silver nanoparticles were characterized using UV-visible spectroscopy and XRD analysis. Lastly, the antibacterial activity of the synthesized AgNPs was evaluated using gram-positive and gram-negative bacterial strains.

In recent years, there has been a major advancement in the synthesis of AgNPs from plants. As a result, it is useful in many fields, including antibacterial, biomedical, industrial, and environmental. Whether for drug delivery, water purification, cancer treatment, drug

delivery, sensing and detection, pollutant degradation, or catalysis. AgNPs have demonstrated promising application opportunities. (Figure 1) illustrates how AgNPs are used in a variety of industries. [31]



**Figure 1: Applications of AgNPs derived from plants in a variety of fields.[31]**

The development of nanoscale iron oxide nanoparticles with remarkable qualities, including stability in physiological settings, biocompatibility, size-dependent magnetic properties, superparamagnetism, high coercivity, high saturation magnetization, and low toxicity [32,33], creates new opportunities in the fields of drug delivery, biosensors, and medicine (Figure 2). According to FDA approval, they are the only kind of magnetic nanoparticles that can be used in clinical settings [42]. Iron oxide nanoparticles have been produced using a variety of physical and chemical synthesis techniques, such as co-precipitation [43], micro-emulsion [44,45], sol-gel [46], and solvothermal [47].



**Figure 2: Applications of FeNPs derived from plants in a variety of fields.**

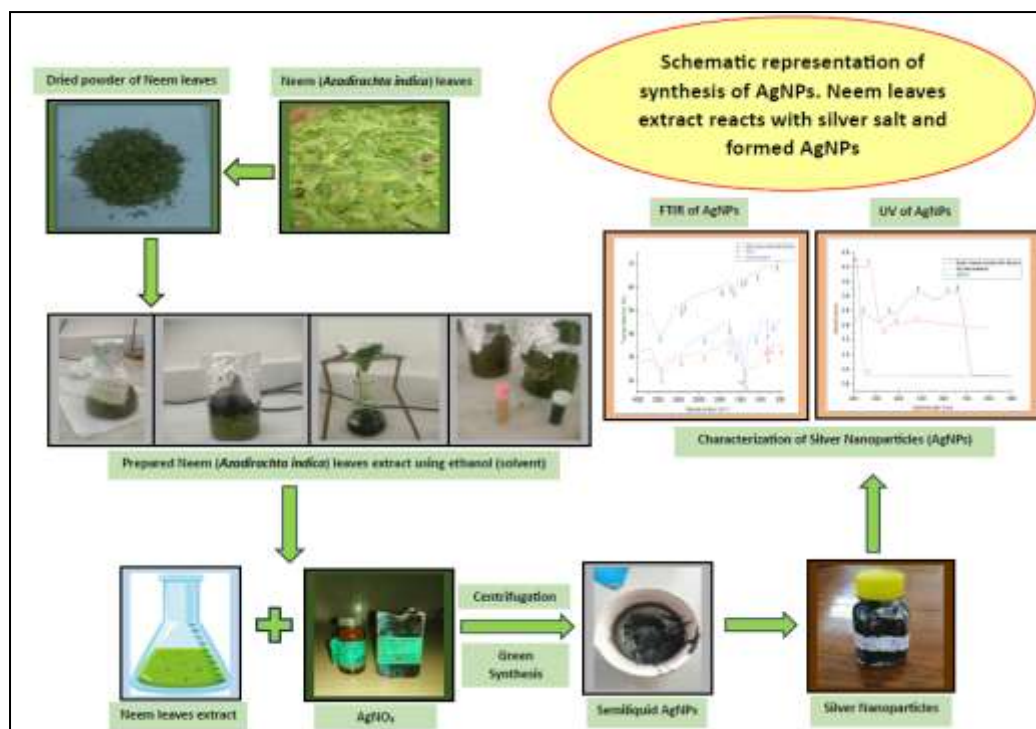
## **2. EXPERIMENTAL**

### **2.1. Materials**

On fresh *Azadirachta indica* leaves that were gathered from Prayagraj, India, a botanist conducted taxonomic identification. Ferric oxide and silver nitrate were obtained from commercial sources and used without additional processing. Throughout the project, double-distilled water was utilized.

### **2.2. Preparation of AgNps**

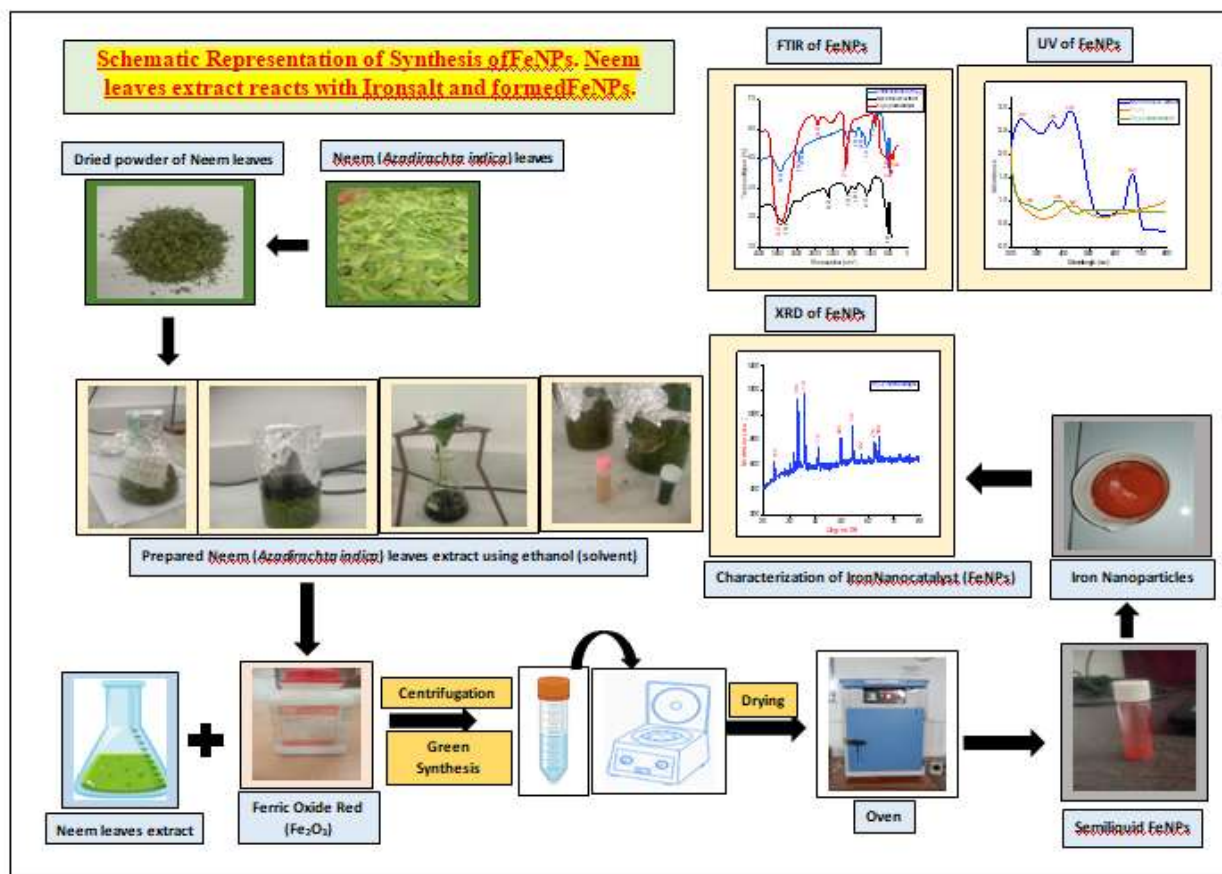
The source of silver nitrate was Sigma-Aldrich Chemical Co. After being cleaned with distilled water, every piece of glassware was dried in an oven. Prior to usage, the agar and petri plates were both sterilized. After boiling 20 g of finely chopped Neem leaves in 100 ml of ethanol for 10 minutes, the mixture was filtered to produce Neem leaf extract. When 45 ml of 1 mM silver nitrate ( $\text{AgNO}_3$ ) was combined with 5 ml of neem leaf extract, a color shift occurred, indicating the formation of AgNPs. (Figure 3)



**Figure 3: Schematic representation of the synthesis of AgNP's. Neem leaves extract reacts with silver salt (AgNO<sub>3</sub>) and formed AgNP's.**

### 2.3. Preparations of Fe nano-catalyst:

The source of ferric oxide was Sigma-Aldrich Chemical Co. After being cleaned with distilled water, every piece of glassware was dried in an oven. Prior to usage, the petri was sterilized. After boiling 20 g of finely chopped Neem leaves in 100 ml of ethanol for 10 minutes, the mixture was filtered to produce Neem leaf extract. A color shift that happened when 5 milliliters of neem leaf extract and 45 milliliters of ferric oxide solution were combined demonstrated the synthesis of FeNPs. (Figure 4)



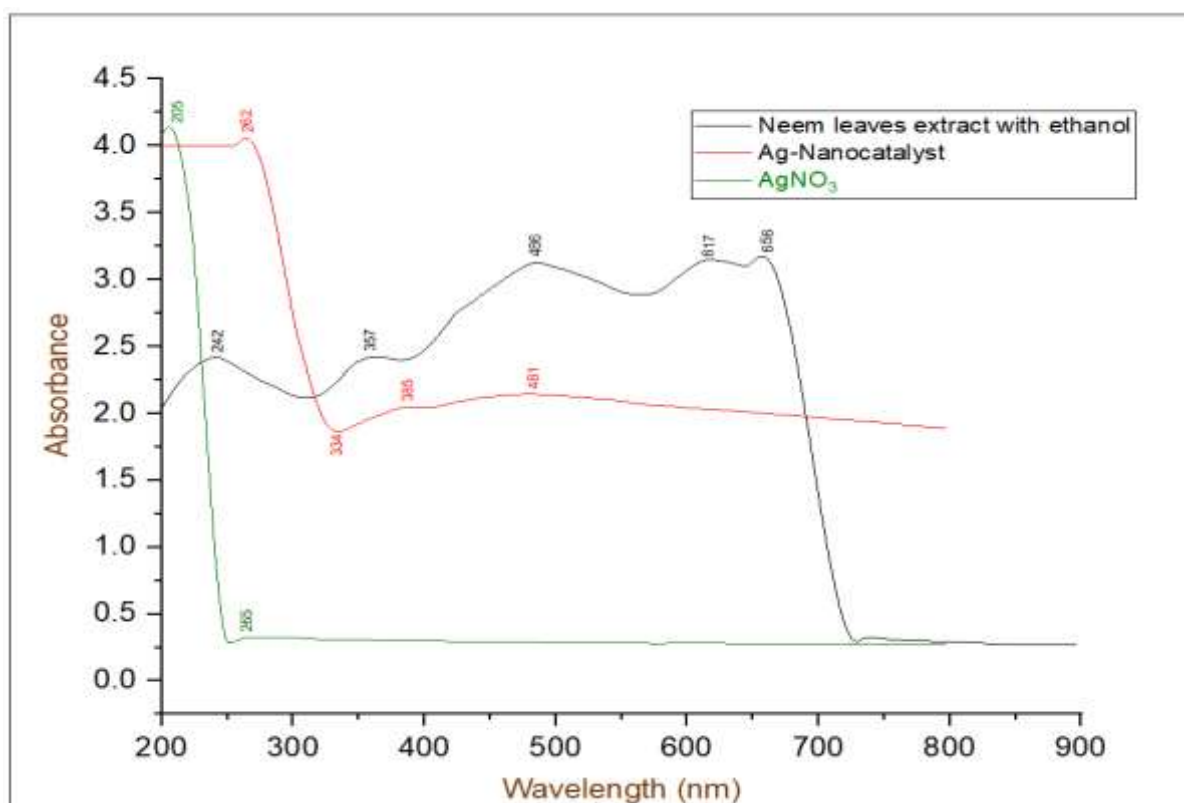
**Figure 4: Schematic representation of the synthesis of FeNP's. Neem leaves extract reacts with ferric oxide and formed FeNP's.**

#### **Characterization Of Prepared Green AgNP's : UV Spectrum of AgNP's:**

UV-vis spectroscopy is a simple yet effective method of determining the properties of produced nanoparticles. AgNPs can interlink with particular light wavelengths due to their photosensitive characteristics. [48] A UV-vis spectrophotometer can be used to characterize a variety of NP morphologies. It provides a thorough, practical, and quick method for characterizing NPs. [49] The proximity of the valence and convection bands permits electron mobility. These freed electrons fluctuate in response to light waves, which is how SPR was created. The degree to which NPs absorb light depends on the chemical environment and particle size. [50,51] A detailed range of AgNPs made from pure plant extract is shown in Figure 5, which is attached below.

UV-vis spectroscopy, which shows that SPR peaks at the same wavelength, has verified for more than a year that AgNPs generated by biological activity are consistent. A complete picture of AgNPs could not be obtained by UV-vis spectroscopy alone.

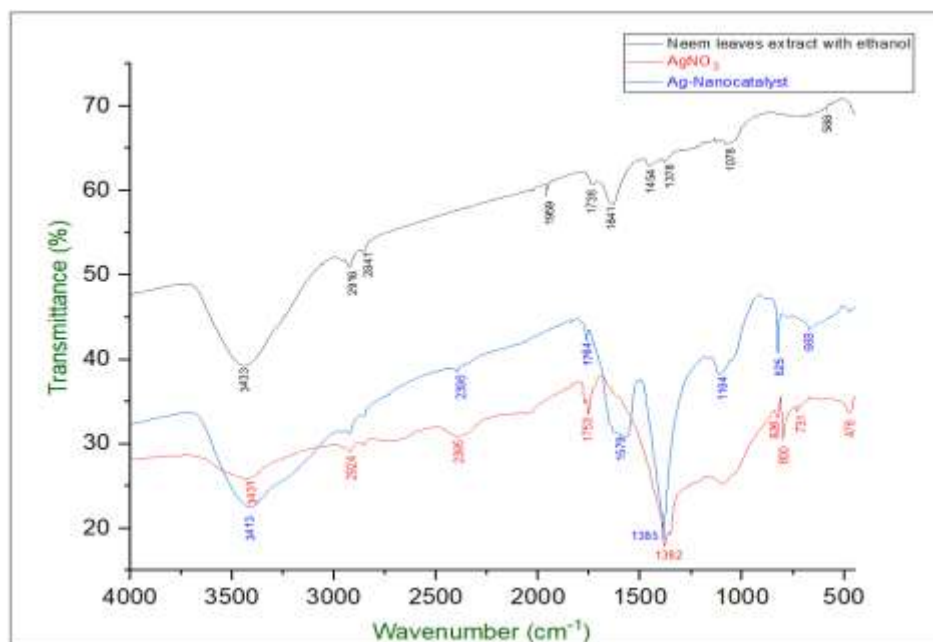




**Figure 5: Characterisation Of Prepared Green AgNP's: UV Spectrum of Silver AgNPs: FT-IR Spectrum of AgNPs green-synthesised using *Azadirachta indica*:**

Using FT-IR spectroscopy, the stability of biosynthesized AgNPs was investigated in relation to the interaction between biomolecules extracted from *A. indica* leaves and AgNO<sub>3</sub> solution. The results are shown in (Figure 6). Stabilized AgNPs from *A. indica* showed absorbance peaks at 3413, 2924, 1579, 1385, 1078, and 508 cm<sup>-1</sup>. The flavones and terpenoids that are abundant in the *A. indica* leaf extract are primarily correlated with these peaks. O-H first emerged at 3413 cm<sup>-1</sup> [52,53], while the C-H stretch of an alkane is represented by 2924. At 1385 cm<sup>-1</sup>, however, symmetrical stretching vibrations of C-C and -COO- were found. At 1078 cm<sup>-1</sup>, the alpha, beta, unsaturated ketone, and ester C-O bond absorption bands were observed.

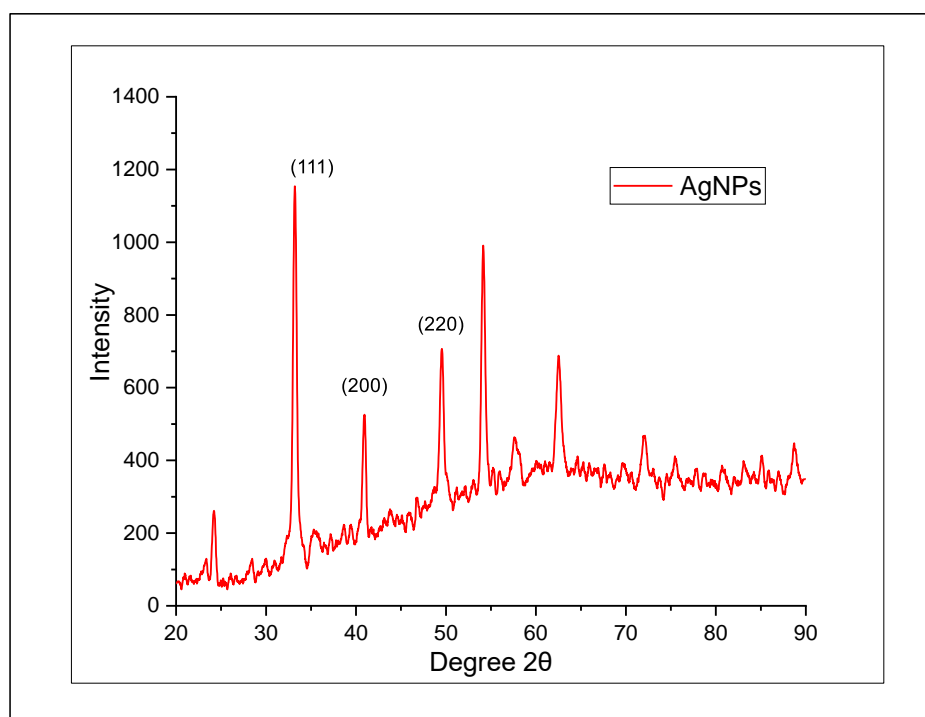
The FTIR spectral study of AgNPs and *A. indica* extract has revealed peak shifts from 3433–3413, 2916–2924, 1641–1764, 1454–1385, 1078–1104, and 508–608 respectively. The changes, as shown in Figure 6, imply that hydroxyl, methyl, carboxylate, and carbonyl functional groups are present in the synthesis and stability of AgNPs. It's possible that interactions between electrons or carbonyl groups caused the flavanones or terpenoids to be adsorbed on the surface of AgNPs. [54] Reducing the sugars in the broth made from *A. indica* leaves may be the reason for the decrease in Ag<sup>+</sup> and the formation of the corresponding metallic NPs. [55]



**Figure6: FT-IR Spectrum of AgNP's green-synthesized using *Azadirachta indica*:**

#### **XRD spectrum of AgNPs green-synthesized using *Azadirachta indica***

The XRD spectrum confirmed the precipitate's crystalline structure as Ag (Figure 7). The lattice planes (111), (200), and (220) are represented by the peak values at  $2\theta = 38.15^\circ$ ,  $44.35^\circ$ , and  $54.59^\circ$  in the face-centered cubic crystal structure of AgNPs. [56,57]



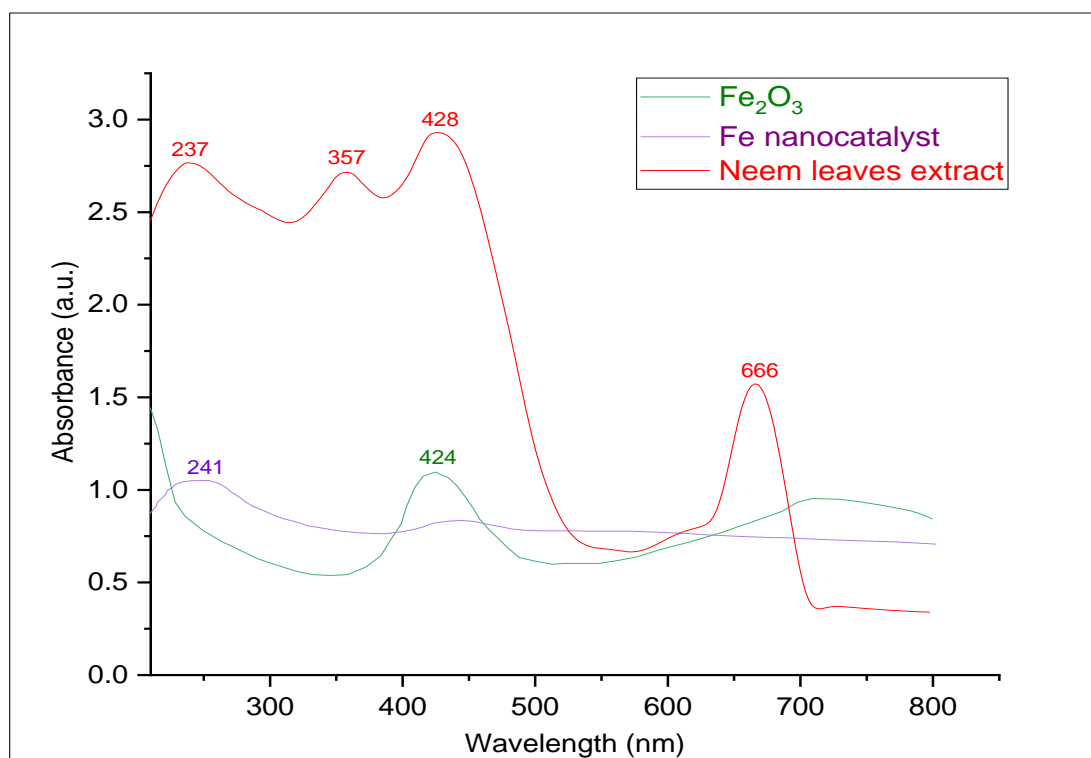
**Figure 7: XRD spectrum of AgNP's green-synthesized using *Azadirachta indica***



### Characterization Of Prepared Green Fe nanocatalyst : UV Spectrum of Fenaocatalyst:

The color of iron salts changed from pale yellow to light orange and then to black when aqueous Neem leaf extract was added. The presence of iron oxide nanoparticles created by the reduction of iron salts was the cause of the solution's color change. When *Azadirachta indica* leaf extract was employed, it was proposed that substances including terpenoids and flavanones functioned as reducing agents [58]. It was observed that the reaction mixture's color changed instantly after roughly ten minutes, after which it did not change again. This showed that all of the iron salts in the reaction mixture had been reduced. After that, the UV-visible spectrum analysis provided additional confirmation of the synthesis of  $\text{Fe}_2\text{O}_3$ -NPs (Figure 8).

The excitation of surface plasmon vibrations in the iron oxide nanoparticles was responsible for the presence of a prominent peak at around 241 nm areas. The combined vibrations of  $\text{Fe}_2\text{O}_3$ -NPs' free electrons in resonance with light waves can account for this. Similar UV-VIS spectra for the synthesis of  $\text{Fe}_2\text{O}_3$ -NPs were reported in works by Hassan et al. (2015) and Devatha et al. (2016) [59,60]. Because neem leaves are non-toxic, this green approach can synthesize  $\text{Fe}_2\text{O}_3$ -NPs in 10 minutes, making it appropriate for biological research and biomedical applications.



**Figure 8: Characterisation Of Prepared Green FeNP's: UV Spectrum of Silver FeNPs:**

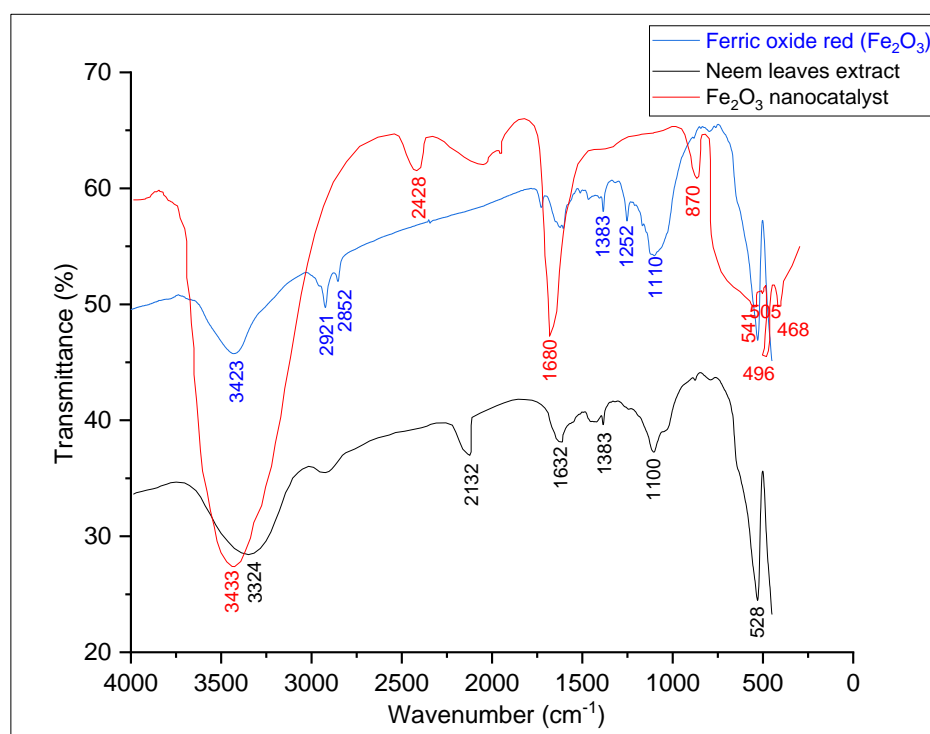
### FT-IR Spectrum of Fenanocatalyst green-synthesised using *Azadirachta indica*:

FTIR analysis was used to determine whether the leaf extract from *Azadirachta indica* contained flavanones and terpenoids, which are responsible for stabilizing iron oxide nanoparticles. (Figure 9) displays the representative FTIR spectra of the produced  $\text{Fe}_2\text{O}_3$ -NPs

and pure *Azadirachta indica* leaf extract. The presence of N-H stretching and bending vibrations of amine groups  $\text{NH}_2$  and OH, as well as the overlap of the stretching vibrations attributable to water and phenolic groups of *Azadirachta indica* leaf extract molecules, are demonstrated by the strong stretching band that appears at around  $3324\text{ cm}^{-1}$  (Figure 9). Following reduction, the intensity drops at  $3433\text{ cm}^{-1}$ , suggesting that *Azadirachta indica*'s phenolic group is involved in the reduction process.

The presence of a  $-\text{COOH}$  group in the *Azadirachta indica* leaf extract was shown by the FTIR spectra's adsorption peak at  $1635\text{ cm}^{-1}$  (Figure 9), which was ascribed to amide  $\text{C}=\text{O}$  stretching. Figure 9's decreasing intensity at  $1685\text{ cm}^{-1}$  indicates that amide  $\text{C}=\text{O}$  stretching is involved in the reduction process. In the meantime, the alkyne group found in phytoconstituents of extracts is represented by the adsorption peak at  $2428\text{ cm}^{-1}$  (Figure 9). Therefore, the existence of these functional groups confirms that molecules of terpenoids or flavanones were chemically bonded to the  $\text{Fe}_2\text{O}_3$ -NPs surface.

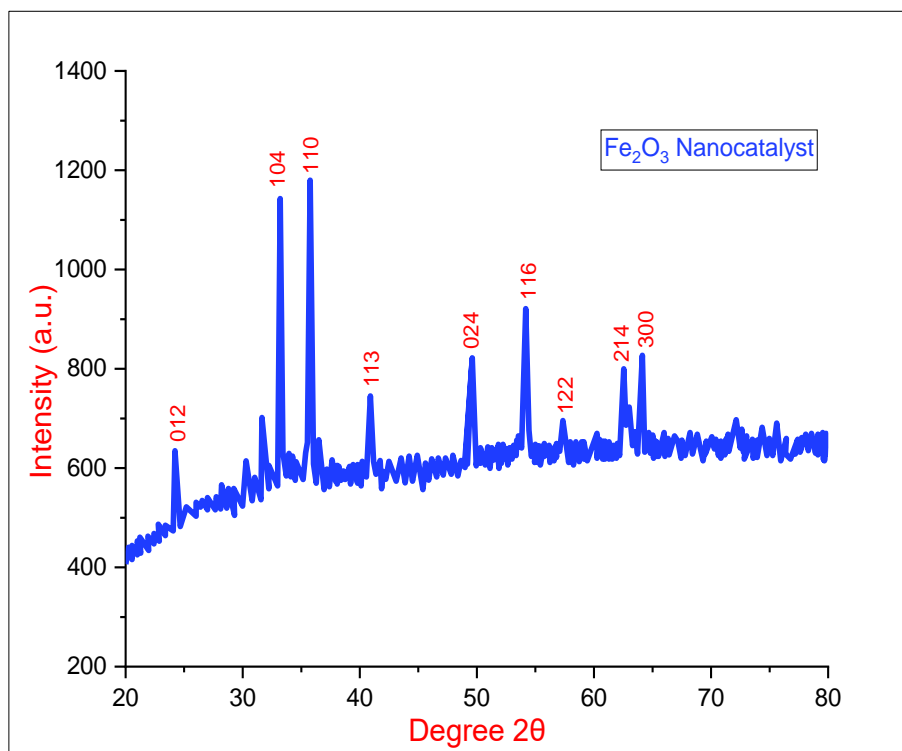
$\text{Fe}_2\text{O}_3$ -NPs have a strong interaction with the flavanones or terpenoids of *Azadirachta indica* leaf extract molecules, as shown by the slight changes in peak position (Figure 9). The presence of the Fe-O stretching band of iron oxide nanoparticles is evident from the appearance of new peaks (Figure 9) at  $541\text{ cm}^{-1}$ ,  $505\text{ cm}^{-1}$ ,  $497\text{ cm}^{-1}$ , and  $468\text{ cm}^{-1}$  [61,62]. The formation of *Azadirachta indica*-mediated  $\text{Fe}_2\text{O}_3$ -NPs in a one-pot reaction was validated by this observation. According to the FTIR findings, the reduction of iron ions that occurs in conjunction with the phenolic compounds in the leaf extract of *Azadirachta indica* may be the plausible mechanism for the creation of  $\text{Fe}_2\text{O}_3$ -NPs [63,64].



**FIGURE 9: FT-IR Spectrum of FeNP's green-synthesized using *Azadirachta indica*:**

### XRD spectrum of AgNP's green-synthesized using *Azadirachta indica*;

The X-Ray powder diffraction measurement was performed at room temperature and a scan rate of  $1^\circ$  per minute in  $0.013^\circ$  steps, covering the  $2\theta$  angle from  $20-80^\circ$ . The XRD pattern of  $\text{Fe}_2\text{O}_3$  showed that six characteristic diffraction peaks of  $\text{Fe}_2\text{O}_3$  were observed at planes  $2\theta = (012)$  at  $25.3^\circ$  ( $104$ ) at  $35.6^\circ$ , ( $110$ ) at  $38.3^\circ$ , ( $113$ ) at  $43.2^\circ$ , ( $116$ ) at  $53.1^\circ$ , ( $122$ ) at  $57.8^\circ$ , ( $214$ ) at  $63^\circ$  and ( $300$ ) at  $65^\circ$ . (Figure 10) The room temperature of the XRD pattern of  $\text{Fe}_2\text{O}_3$  was plotted by Rietveld refinement using GSAS and EXPGUI software [65,66].

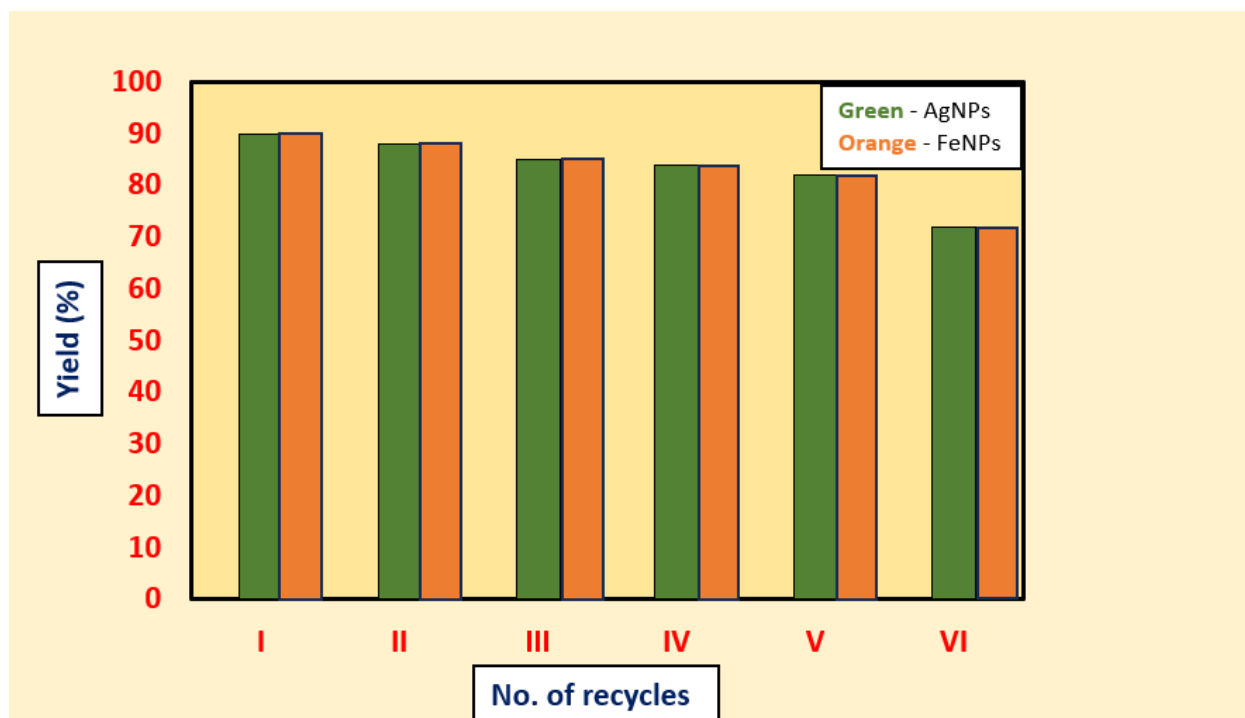


**Figure 10: XRD spectrum of FeNP's green-synthesized using *Azadirachta indica***

### RECYCLABILITY OF AgNps and Fe nanocatalyst:

To investigate the recyclability and regeneration of the green biosynthesized AgNps and FeNps, it was isolated from the solution. The green biosynthesized AgNps was rinsed with acetone and dried for 30 minutes at room temperature after each experiment. The recovered AgNps and FeNps was found to be reusable in five reaction cycles without losing its selectivity or activity. Finally, to uniqueness and advantage of biosynthesized AgNps and FeNps by *Azadirachta indica* leaves extract.

FeNp powder was vigorously stirred while soaking in acetone for an entire night following each treatment. After that, the particles were gathered, cleaned, and centrifuged multiple times using ethanol, distilled water, and acetone before being left to dry overnight at  $120^\circ\text{C}$  in an oven. For five cycles, powder FeNps were recovered without losing their effectiveness.



**Figure11: Recyclability of AgNps and FeNps.**

## Conclusion

As a green, environmentally friendly reducing and capping agent, *Azadirachta indica* (neem) leaf extract was used to create silver nanoparticles (AgNPs). The color shift from greenish brown to dark brown verified the formation of AgNPs. The biosynthesized AgNPs were characterized using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and UV-Vis spectroscopy. A distinctive surface plasmon resonance peak was visible in the UV-Vis spectrum, and functional groups like hydroxyl, methyl, carboxylate, and carbonyl were detected by FTIR analysis. These findings suggested that flavonoids and terpenoids were involved in the reduction and stability of AgNPs. The crystalline nature of the produced AgNPs with a face-centered cubic structure was validated by XRD examination. Both gram-positive and gram-negative bacterial strains were susceptible to the AgNPs' possible antibacterial action.

In this study, we successfully synthesized iron oxide nanoparticles using *Azadirachta indica* as a one-pot green method. The color shift that was seen right away implied that the development of iron oxide nanoparticles was indicated by the creation of a black solution. The effectiveness of *Azadirachta indica* extract as a reducing and stabilizing agent was demonstrated by the quick reduction process. The cubic crystal structure of impurity-free iron oxide nanoparticles was revealed by the XRD pattern. The interactions between *Azadirachta indica* and iron oxide nanoparticles were demonstrated by FTIR.

It has the potential to be used in biomedical applications and will soon play a significant role in magnetic targeting drug delivery. The achievement of such quick time scales for iron oxide

nanoparticle synthesis could be a competitive alternative to the traditional chemical protocols and a low-cost candidate as reductant for iron oxide nanoparticle synthesis.

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