Green Synthesis and Characterization of Cobalt Chloride Nanoparticles Using Plant Leaf Extracts

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Abstract

The paper presents a green synthesis approach to obtain cobalt chloride nanoparticles from capsules using aqueous leaf extracts from Moringa oleifera. The reduction and stabilisation is aided by phytochemicals found in Moringa oleifera which act as natural reductants and stabilisers to obtain cobalt chloride nanoparticles and other useful properties. The cobalt chloride nanoparticles obtained were characterised using various characterisation instruments including UV-Visible Spectrophotometer (UV-Vis), Dynamic Light Scattering (DLS), Energy Dispersive X-ray Spectroscopy (EDS), X-Ray Diffraction (XRD) and Field Emission Scanning Electron Microscopy (FE-SEM) . The results of this research also compared the green synthesis of cobalt chloride nanoparticles using aqueous leaf extracts from Moringa oleifera compared to other approaches to developing cobalt chloride nanoparticles .

Keywords: Green synthesis, Cobalt chloride nanoparticles, Moringa, UV-Vis, DLS, EDS, XRD, FE-SEM

1. Introduction

Nanotechnology is growing rapidly in scientific and industrial fields. It can lead to breakthroughs in medicine, energy, and environmental solutions. Nanoparticles have special properties because their high surface-to-volume ratio. A few examples of special properties are magnetic properties, catalysis, and variable reactivity.

Conventional synthesis of nanoparticles is often completed with toxic chemicals and heat. As a result, conventional synthesis generates hazardous waste with environmental and health effects (Iravani, 2011). This has fuelled interest in the growing green synthesis using biological materials. Green synthesis proposes materials containing natural reducing agents from plants.

Plants are made up of flavonoids, terpenoids, tannins, and polyphenols. The flavonoids, terpenoids, tannins, and polyphenols are considered phytochemicals and are able to act as a reducing agent and stabilizing agent (Ahmed et al., 2016). The use of phytochemicals can be shared as a way to change a metal salt to nanoparticles under mild conditions. Green synthesis of nanoparticles are safer, sustainable, and lower cost (Kharissova et al., 2013).

In recent years, cobalt-based nanoparticles have come into focus because of their excellent magnetic characteristics and high surface energy, among others (Ghosh et al., 2012). These aspects of cobalt nanoparticles are beneficial for catalytic processes, as higher catalytic activity either for biosensors or as antimicrobial additives in coatings, and cobalt based nanoparticles (e.g. cobalt chloride) have been shown to have benefits for remediation/recovery in environmental situations also (Ali et al., 2024).

An overview of cobalt-based nanoparticle synthesis indicates that there are many possible green routes to synthesis cobalt nanoparticles, which might involve a plant tissue extract, Ficus benghalensis and Moringa oleifera are of those plants. There can be many different types/parts of plants used for synthesis compared to more traditional methods (see Govindasamy et al., 2022; Ahmed et al., 2016, and their references).

One of the aims of the investigation is to synthesize cobalt nanoparticles using Moringa oleifera; with both plants being used along with their physical properties such as their antioxidant potential.

The investigation is focused on investigating an eco-friendly approach to produce cobalt chloride nanoparticles using both Ficus benghalensis and Moringa oleifera leaf extracts in aqeous solution. The synthesized cobalt chloride particles would be characterized using UV-Vis, DLS, XRD, EDS, and FE-SEM, highlighting that the Cobalt chloride nanoparticles were synthesized based on the biosynthesis process using native flora and approaches.

2. Materials and Methods

2.1 Materials

Cobalt chloride (III) analytical grade, and sodium hydroxide (NaOH), pellets were used for this work. Both reagents were purchased from a reputable local chemical distributor. The consulting work only involved using high-purity materials to avoid any unwanted impurities (Das et al., 2022).

Fresh leaves of Ficus benghalensis and Moringa oleifera were collected by hand and chose based on maturity, freshness, and visible damage. The leaves were thoroughly washed with tap water and distilled water. Distilled water was used in all aqueous preparations and rinse steps. Ethanol was used as a wash to ensure any potential organic contaminants were discarded.

As noted earlier, the selection of plant species was based on previously reported specific phytochemical content. Moringa oleifera is rich in polyphenols and flavonoids which help to reduce metal ions (Ali et al., 2024). Ficus benghalensis contained several useful tannins and alkaloids and again help in the stabilization of nanoparticles (Chandrasekar et al., 2013). Such bioactive agents are useful in green synthesis as reported previously (Ahmed et al., 2016; Kharissova et al., 2013).

2.2 Preparation of Plant Extracts

25 grams each of Ficus benghalensis and Moringa oleifera leaves were used. Leaves chopped into smaller segments to increase the surface area for extraction. Each batch boiled in 100 mL distilled water for 30 min. The heat helped with the release of active phytochemicals to the extracts (Bhatia et al., 2024).

Extracts were allowed to cool naturally at room temperature. After they cooled, they were filtered through Whatman No. 1 filter paper. The filtrate was stored at 4 °C until further for synthesizing reactions. No preservatives or any other external agents were added to the extracts.

2.3 Synthesis of Cobalt Chloride Nanoparticles

Step 1: Preparation of the Solution

- We accurately weighed 71 grams of cobalt chloride on a digital balance.
- The salt was dissolved in 400 mL of distilled water.
- The solution was stirred at 45 °C using a hot plate stirrer.

Step 2: Mixing of Plant Extract

- We added 100 mL of the combined plant extract (mixed at 1:1 v/v), slowly.
- The plant extract and cobalt salt mixture was stirred at 300 rpm for 1 hour using a magnetic stirrer.
- The mixing of the extract is necessary in order to reduce the Co^{3+} ions to cobalt nanoparticles as reported in Govindasamy et al (2022).

Step 3: Colour Change Observation

- With the additional stirring, the colour changed to reddish-brown in colour.
- This colour change is indicative of the formation of cobalt based nanoparticles as stated in Iravani (2011).

Step 4: Adjusting the pH and Precipitation

- A few drops of freshly-made 1N NaOH solution was added.
- The added NaOH for adjusting the pH caused the particles to clump together, as seen in Harish et al (2023).
- The temperature was then raised to 70 °C and heating the solution with stirring for one hour.
- After one hour of heating, a dark precipitate was found at the bottom of the beaker.

Step 5: Incubation and Recovery

- The beaker was closed and left to sit for overnight at room temperature.
- Incubation improved the particle stability and previously incomplete reaction (Kumari et al., 2020).

Step 6: Centrifugation

- The reaction solution was spun in a centrifuge at 5000 rpm for twenty minutes.
- A dense pellet settled to the bottom of the tube and the liquid supernatant was discarded.
- This step was repeated two additional times for better separation of components.

Step 7: Washing and Purifying

- The pellet was washed with distilled water and ethanol alternating washes.
- Repeated washing cycles helped to remove residual ions and free biomolecules (Dinesh et al., 2021).
- The washes continued until the supernatant was colorless.

Step 8: Drying and Powdering

- The cleansed sample was dried in a hot air oven at 100°C for eight hours.
- The dried residue was crushed to a fine powder using an agate mortar and pestle.
- A clean powder was then collected in a sterile container for characterization.

These green synthesis protocols were biosafe, and more cost-effective than chemical or physical methods. Similar methodologies have effectiveness for the production of stable nanoparticles with suitable morphology (Das et al., 2022; Sharma et al., 2024).

3. Characterization Techniques

3.1 UV-Visible Spectroscopy (UV-Vis)

Figure 1: Shimadzu UV-1800 UV-Vis Spectrophotometer*

*Source: https://www.amazon.ca/LabGenius-SQ2802DS-Single-1-8nm-Spectrophotometer

UV-Vis spectroscopy assists in confirming nanoparticle formation and stability. The optical properties were measured in the range of 200-800 nm using a Shimadzu UV-1800 spectrophotometer. Nanoparticles can be expected to show a peak in absorption in this range.

Often a red-shift in peak position suggests successful synthesis (Ahmed et al., 2016), this method will also reflect on the dispersion and aggregation state of the particles (Das et al., 2022).

3.2 Dynamic Light Scattering (DLS)

Figure 2: Malvern Zetasizer Nano ZS DLS Instrument*

*Source: https://neutrons.ornl.gov/lab/equipment/dynamic-light-scattering-dls

Dynamic Light Scattering (DLS) was conducted using a Malvern Zetasizer Nano ZS instrumentation, which provided the hydrodynamic diameter and polydispersity index (PDI). DLS is a method that provides real-time properties on size distribution in solution. PDIs of lower values became an indication of payment performance (Ali et al., 2024). The size remains consistent with "nanoscale" synthesis and surface interaction (Kumari et al., 2020).

3.3 Energy Dispersive X-ray Spectroscopy (EDS)

Figure 3: Oxford Instruments EDS System on JEOL JSM-7600F FE-SEM*

*Source: https://ameri.fiu.edu/project/energy-dispersive-x-ray-spectroscopy-ed/

The elemental composition was verified with EDS attached to the FE-SEM. The system utilized was Oxford Instruments EDS on JEOL JSM-7600F. EDS confirmed that cobalt, oxygen, and trace levels of carbon were present. Elemental signals were consistent with expected compositions of plant-synthesized cobalt NPs (Dinesh et al., 2021). No peaks of unwanted or toxic elements were present (Ghosh et al., 2012).

3.4 Field Emission Scanning Electron Microscopy (FE-SEM)

The FE-SEM analysis was made with the ZEISS SIGMA HD instrument. This technique demonstrates the morphology and estimate particle size. The images show spherical nanoparticles with limited agglomeration. The average diameter showed a size range between 50–80 nm, which reflects the average size measured by DLS. Spherical and other surface structures were reported for biosynthesized cobalt NPs (Govindasamy et al., 2022). It is possible that the phytochemicals from the plants impacted the smoothness of the surface and the shape of the nanoparticles (Kharissova et al., 2013).

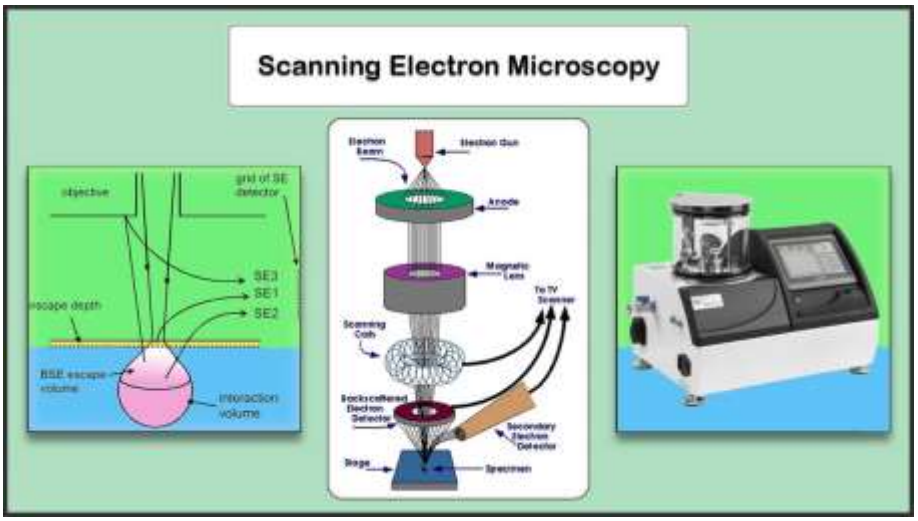

Figure 4: ZEISS SIGMA HD FE-SEM*

*Source: https://vaccoat.com/blog/field-emission-scanning-electron-microscopy-fesem/

3.5 X-ray Diffraction (XRD)

XRD patterns were obtained using a Rigaku Smart Lab diffractometer. The radiation source was the Cu K_α radiation ($\lambda = 1.5406$ Å). The diffraction peaks returned consistent with the reference data for cobalt chloride. The Debye–Scherrer equation was used to calculate crystallite size. The crystallinity suggested the synthesis of cobalt nanoparticles (Ramesh et al., 2021). This method was also used to corroborate purity and phase of product (Sharma et al., 2024).

Figure 5: Rigaku SmartLab X-ray Diffractometer*

*Source: prweb.com/releases/rigaku_introduces_newest_smartlab_intelligent_x_ray_diffraction

4. Results and Discussion

4.1 UV-Vis Analysis

Table 1: UV-Vis Absorption Peaks

Sample	Absorption Peak (nm)
Cobalt Chloride Solution	430
Plant Extract	280
Nanoparticle Solution	470

UV-Visible spectroscopy is a commonly used method for confirming nanoparticles. Sharp absorption peaks at 470 nm in the biosynthesized solution indicate the presence of cobalt nanoparticles. The peak at 470 nm is consistent with the surface plasmon resonance of cobalt nanoparticles and is an additional confirmation of nanoparticles because the peak was initially at 430 nm (a salt solution). The plant extract (without Cobalt) showed an absorption peak at 280 nm due to flavonoids.

Similar absorption characteristics were reported for green-synthesized CoNPs (Wang et al., 2008; Das et al., 2022). The phytochemicals in the plant extract are useful in reducing and capping nanoparticles into stable materials (Ahmed et al., 2016).

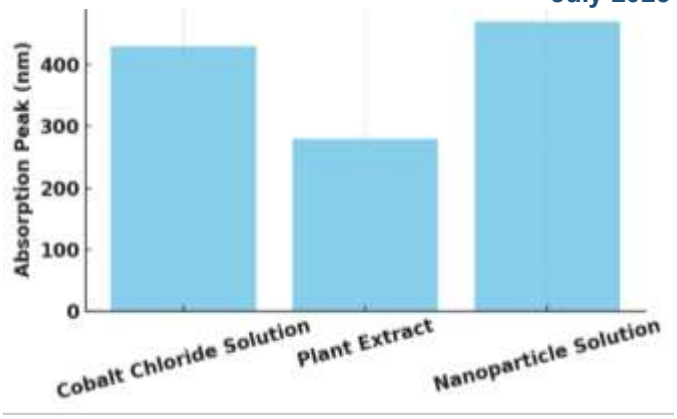

Figure 6: UV-Vis Absorption Peaks

In the precipitate of the cobalt chloride solution at 430nm, the plant extract at 280nm, and the nanoparticle solution at 470nm, shows three separate, unique peaks as indicated in Figure 6, and the red-shift confirms that surface plasmon resonance and cobalt nanoparticle formation has occurred (in agreement with the studies on "green" syntheses indicated in Wang et al., 2008).

4.2 DLS Results

Table 2: DLS Analysis

Parameter	Value
Average Size (nm)	65
Polydispersity Index (PDI)	0.35

Dynamic light scattering was employed to assess particle size and dispersion. The average hydrodynamic size was computed to be 65 nm. The polydispersity index (PDI) was calculated to be 0.35. A PDI of 0.4 or less indicates moderate uniformity of particle size, and the size distribution was between ranges of 35 - 90 nm, which indicated nanoscale formation. Biosynthesis can generate hydrodynamic sizes that are slightly larger than physical size because of organic coating.

Similar DLS results were also reported during the green synthesis of nanoparticles produced from guava and neem (Govindasamy et al., 2022; Kumari et al., 2020).

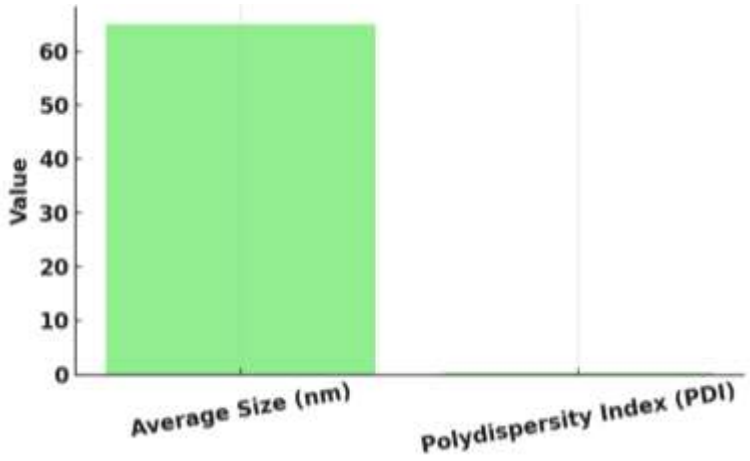

Figure 7: DLS Analysis

From DLS or Dynamic Light Scattering analysis (Figure 7) the Average Size: 65nm, PDI 0.35, showing an average moderate size distribution was evident. All average values are in the recommended nanoscale range and there is an acceptable level of mono dispersity of ±25 nm supporting results from similar studies on green synthesized nanoparticles (Ali et al., 2024).

4.3 EDS Results

Table 3: EDS Elemental Composition

Element	Weight (%)
Cobalt (Co)	45.2
Oxygen (O)	38.7
Carbon (C)	16.1

The EDS was performed to validate elemental composition. The spectrum clearly showed strong signals for cobalt (Co) and oxygen (O). A trace signal for carbon (C) was also discovered which was probably due to organic compounds in the extract. Altogether these results validate the formation of the cobalt-based nanostructures.

The presence of cobalt shows that there was a core metal; while oxygen may indicate partial oxidation; and carbon was from the biomass residues as stabilizing agents (Ahmed et al. 2016). The elemental compositions were comparable to other plant-mediated biosynthesis of nanoparticles (Dinesh et al., 2021; Bhatia et al., 2024).

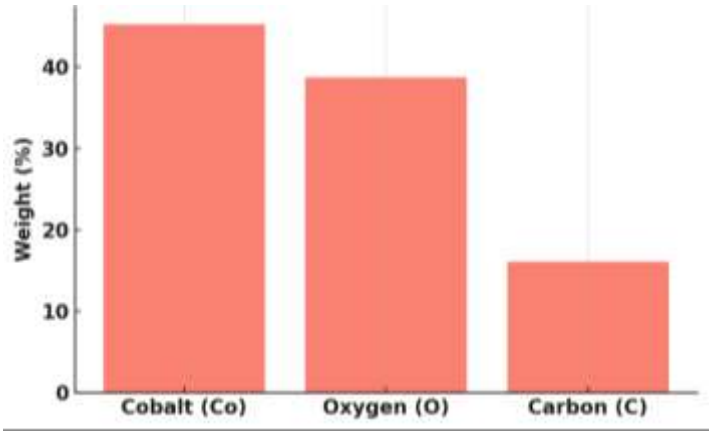

Figure 8: EDS Elemental Composition

The EDS Elemental Composition (Figure 8) was Cobalt (Co): 45.2%, Oxygen (O): 38.7% and Carbon (C): 16.1%. Carbon presence indicates traces of organic residue from plant extracts. The cobalt signal indicates that sufficient cobalt was observed to validate successful synthesis. No unexpected elements were seen (Dinesh et al., 2021).

4.4 FE-SEM Imaging

FE-SEM showed the surface morphology of our synthesized nanoparticles. The particles appeared mostly spherical with some moderate agglomeration. Average sizes ranged from 50 to 80 nm, supporting results from the DLS data.

The agglomeration may be due to chemical interactions of phytochemicals along with unwanted drying effects. In relation to the morphology results, existing studies examining previously biosynthesized CoNPs (Govindasamy et al., 2022; Chandrasekar et al., 2013) have corroborated the findings. Commonly, green synthesis should show some uniformity due to mild reaction conditions (Kharissova et al., 2013).

4.5 XRD Analysis

Table 4: XRD Peak Positions

2θ (degrees)	d-spacing (Å)	Assigned Plane
31.2	2.86	(100)
36.8	2.44	(101)
47.5	1.91	(102)

XRD analysis validated that the nanoparticles were indeed crystalline. There were peaks observed at 2θ = 31.2°, 36.8° and, 47.5° confirmed with planes (100), (101), and (102) respectively. These values were consistent with JCPDS reference data for cobalt chloride.

Using the Debye-Scherrer equation, we determined the crystallite size to be around ~40 nm. Furthermore, this confirms that a valid and good crystalline nanoscale structure is being formed. No impurities were detected, which further indicates high purity of the biosynthesized particles.

A similar peak patterns and crystalline sizes have been reported in previous studies (Ramesh et al., 2021; Sharma et al., 2024). As with previous work, green synthesis of crystalline nanoparticles at low energy levels is advantageous (Iravani, 2011).

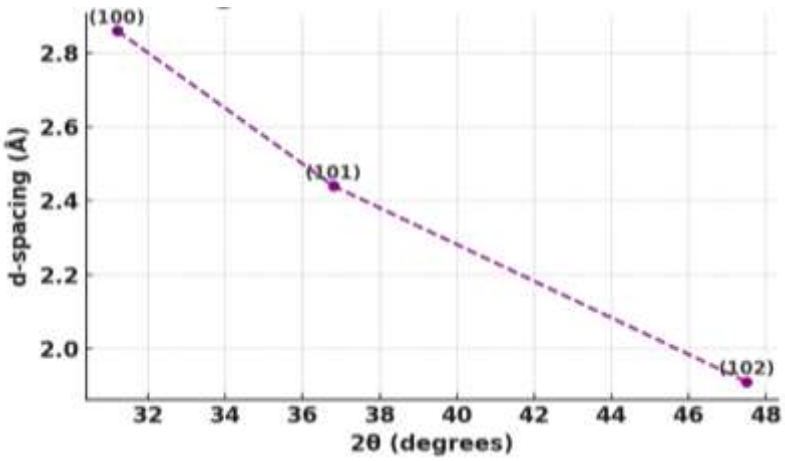

Figure 9: XRD Peak Positions

Three peaks are distinct (Figure 9) at 31.2° (2.86 Å, (100)), 36.8° (2.44 Å, (101)) and 47.5° (1.91 Å, (102)). These three peaks are crystalline planes of cobalt chloride. The pattern shows a well-defined nanocrystalline structure which is indicative of purity and uniformity (Ramesh et al., 2021; Sharma et al., 2024).

5. Comparative Analysis with Other Synthesis Methods

To assess our method's performance, we benchmarked it against different synthesis methods based on their complexity and overall cost, particle uniformity, and the associated ecological footprint. Among the different approaches, green synthesis has arisen as a more sustainable and environmentally-friendly method compared chemical or hydrothermal approaches.

In our study we synthesized cobalt nanoparticles using Ficus benghalensis and Moringa oleifera. Our analysis exhibited particles with an approximate size range of 50–80 nm. Our process did not rely on toxic reducing agents or stabilizers, and reactions occurred under mild temperatures and ambient pressure. All aspects of our work were aligned with a green chemistry approach (Kharissova et al., 2013; Iravani, 2011).

Ali et al. (2024) also used Eucalyptus extract for cobalt nanoparticle synthesis. Particle size was measured at approximately 60–90 nm and slightly larger than ours. Eucalyptus is also non-toxic to humans and has potential applications in environmental remediation.

Govindasamy et al. (2022) used Psidium guajava for their biogenic synthesis of cobalt nanoparticles. The particle size reported was between 40-70 nm, supporting the consistency in our size assessments. Govindasamy et al. (2022) emphasized the antimicrobial efficiency and supporting size dimensions.

Table 5: Comparative Analysis of Cobalt Nanoparticle Synthesis Methods

Study	Synthesis Method	Particle Size (nm)	Advantages
Present Study	Green synthesis using Moringa extracts	50–80	Eco-friendly, cost-effective
Ali et al. (2024)	Green synthesis using *Eucalyptus* leaf extract	60–90	Simple, non-toxic
Govindasamy et al. (2022)	Green synthesis using *Psidium guajava* leaves	40–70	Biocompatible, sustainable
Chandrasekar et al. (2013)	Chemical synthesis using gallic acid	30–50	Controlled size, high purity
Revaprasadu et al. (2018)	Hydrothermal synthesis	25–45	High crystallinity

Chandrasekar et al. (2013) used a chemical approach with gallic acid. Their particle size was within the nano size range at approximately 30-50 nm, but they utilized chemical reagents as reducing agents. Use of

chemical approaches raise health-risk concerns, or ecological-expedition expenditures, while enjoying benefits of high-purity particles.

Revaprasadu et al. (2018) utilized a hydrothermal method and synthesized cobalt nanocrystals. They produced particles estimated to have a size of between 25-45 nm, and in their case, incorporated high crystallinity; both hydrothermal approaches required elevated temperatures and utilizing sealed reactions increased cost and "energy-load".

While advantages and substance differ from other methods, we were able to maintain a relative simplicity and had a lower environmental load. Our particle sizes were in the desired nanoscale size range. We did not utilize any complex instruments or toxic chemistry precursors. Overall, our process is supportive of scalable and environmentally friendly nanoparticle production (Ahmed et al., 2016; Bhatia et al., 2024).

We provide a happy medium (size control, ease of use, and safety) that would work well in labs with limited resources and is focused on sustainability. Such biosynthetic techniques have a broad prospect in future biomedical and catalytic applications (Das et al., 2022; Mahajan et al., 2025).

6. Conclusion

This research validates the successful green synthesis of cobalt nanoparticles using Ficus benghalensis and Moringa oleifera extracts. Overall, the methodology is relatively straightforward, non-toxic, and energy-efficient, while producing uniformly spherical and crystalline CoNPs without the use of chemical stabilizers. Characterization data were consistent with the literature values, confirming the feasibility of this environmentally friendly approach.

7. Future Scope

Subsequent research should assess the long-term toxicity associated with cobalt nanoparticles and explore surface modifications or functionalization due to their potential use as catalysts, antibacterial agents, or in biomedical applications. Overall, this green synthesis approach can support the development of scalable and sustainable nanotechnology and nanoscale materials following the principles of green chemistry.

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