OPEN ACCESS

Saudi Journal of Biomedical Research

Abbreviated Key Title: Saudi J Biomed Res
ISSN 2518-3214 (Print) |ISSN 2518-3222 (Online)
Scholars Middle East Publishers, Dubai, United Arab Emirates
Journal homepage: https://saudijournals.com

Review Article

Phytogenic Production of Monodispersed Nickel Nanoparticles: A Mechanistic Approach on *Pyrus calleryana*

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DOI: https://doi.org/10.36348/sjbr.2025.v10i06.005 | **Received:** 19.05.2025 | **Accepted:** 24.06.2025 | **Published:** 28.06.2025

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Abstract

In the present study, a eco-friendly, low cost and biocompatible route to synthesize the NiNPs, has been proposed using the *Pyrus calleryana* (Callery pear) leaves extract, which is comprised of polyphenols, flavonoids and reducing sugar, which act as an excellent reducing and stabilizing agent. The as prepared NiNPs were characterized by various methods such as X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIF), atomic force microscopy (AFM), and UV-Vis spectroscopy. XRD proved crystalline tetragonal structure of NiNPs and SEM and AFM showed the homogeneous distribution of nanoparticles with particlesized ranging between 18.75 - 32.43 nm. Functional groups (hydroxyl, carbonyl and aromatic rings) essential in nanoparticle stabilization were characterized with FTIR, whereas UV-Vis, evidenced an absorption peak at ~380 nm, corresponding to NiNP formation. The phytochemical composition of the *P. calleryana* extract was an important factor for nanoparticle synthesis as polyphenols and flavonoids served to nucleate and prevent aggregation. This research featured the potential of plant-assisted synthesis as a green approach for nano-synthesis and provided structural and functional attributes of bio-mediated NiNPs for environmental and industrial applications.

Keywords: Green synthesis, Nickel nanoparticles, *Pyrus calleryana*, Phytochemicals, Catalytic applications, Nanotechnology.

INTRODUCTION

Nanotechnology is one of the promising fields for material science which is expected to offer new remedies in medicine, catalysis, environmental remediation and etc., (Khan *et al*., 2023). Among the metallic NPs, nickel NPs (NiNPs) have emerged as a NPs of interest due to their unique magnetic, catalytic and antimicrobial properties (Yousif *et al*., (2024)). Conventional chemical route of synthesis is toxic and requires chemical reducing agents, hence green synthesis by plant extracts has emerged as one of the eco-friendly, cost effective and biocompatible option now (Makarov *et al*., 2022).

Pyrus calleryana (Callery pear), a polyphenol, flavonoid, and reducing sugars rich plant and which have potential applications for reducing and stabilizing agent in nanoparticle (Li *et al.,* 2023). Novel studies has also shown that the method for preparing monodispersed NiNPs is efficient, and the stability and bioactivity of the chemically prepared NiNPs and AuNiNPs are higher compared with chemically prepared NiNPs (Wang *et al.,* 2023). The P. calleryana phytochemicals has played the dual role of reduction and capping agent preventing aggregation of the particles and binding of the Nis^{+2}-cations (Zhang *et al*., (2023). It fulfills the aspects of green chemistry by lessening the production of dangerous waste products and energy usage (Varma, 2023; Ghidan & Antary, 2023).

In this paper, we report the effective synthesis of NiNPs by P. calleryana leaf extract and the study of its structural, optical and catalytic properties by the help of sophisticated techniques (such as XRD, SEM, XRD, FTIR). The results are projected to provide a foundation for green synthesis of nanomaterials effective in wastewater treatment and for renewable energy purposes.

MATERIAL AND METHODS

The leaves of *Pyrus calleryana* were collected from trees of Al-Rashidiya at Baghdad, Iraq. After collection, washed in succession, with tap water to remove the gross contamination followed by deionized water to cleanse finally.

Preparation of Cold Alcoholic *Pyrus calleryana* leaves Extract:

A cold ethanolic extract was obtained by macerating 100 g of citrus leaves in 600 mL of 70–80% ethanol for 24 h at room temperature with intermittent shaking. The suspension was then filtered successively through Whatman No. 1 filter paper and a Buchner funnel lined with sterile gauze to remove particulates. The extract was clarified by centrifugation of the filtrate at 2500 rpm for 15 min. The supernatant was recovered and then concentrated under evaporation in an incubator at 40–45°C for 48–72 h, until a dry extract was obtained. The extract was finally cooled to 4°C and kept refrigerated until use.

Prepare a 1 M Ni stock solution by dissolving 1.9 g of nickel(II) nitrate hexahydrate ($Ni(NO_3)_2 \bullet 6H_2O$) in 10 mL of distilled water, and stir the solution.

To 50 mL volumetric flask add 45 mL distilled water and 5 mL 1 M nickel stock solution to prepare 0.1 M nickel solution. Dilute nickel solution (0.1 M) 50 mL is mixed with 50 mL plant extract (Pyrus calleryana leaves extract which acts as reducing and stabilizing agent). The resultant solution is thoroughly stirred for 1-2 hr at 60-80°C in order to form nanoparticles. Neutralize the pH to ~10 at 1 M NaOH and precipitate of nickel hydroxide (Ni(OH)2).

The precipitate is centrifuged (10,000 rpm, 15 min), washed with distilled water/ethanol, and then dried at 80°C for 12 h. The dry powder was transferred to a furnace and calcined at 400–500 °C for 2 h to obtain crystalline NiO NPs.Khan, S. A., Shahid, S., & Lee, C.-S. (2023). Plant-mediated synthesis of nickel oxide nanoparticles.

Fourier-Transform Infrared Spectroscopy (FTIR)

The FTIR spectra was recorded using a FTIR spectrophotometer (NICOLET Magna-IR 550) with a resolution of 4 cm −1. The FTIR spectra represent a direct consequence of the fact that vibrational energy is absorbed for molecular structure characterization (Smith & Dent, 2019). This method works on the fact that the presence of functional groups is detected by observing the characteristic frequency of infrared absorption of functional groups in the region 4000-400 cm^{-1} (Abdul-Razaq& Ahmed2024). The approach yields qualitative and quantitative information with clear spectral fingerprints for molecular composition and phase attributes (Stuart, 2020). Nanoparticles Phenomenons being investigated in recent years include nanoparticle characterisation and polymer analysis.

Atomic Force Microscopy (AFM)

AFM is based on direct tip-sample interactions that result in a three-dimensional image of the sample surface with atomic-scale resolution (Eaton & West, 2020). This scanning probe method reaches <1 nm vertical and 5 nm lateral resolution, allowing for accurate determination of surface roughness (Meyer *et al*., 2021). Unlike EM, AFM does not require vacuum conditions, and can be used with a variety of materials, such as biomolecules and nanomaterials.

UV-Visible Spectroscopy

UV-Vis spectroscopy investigates the optical properties by electronic transitions, specifically in the case of surface plasmon resonance in NPs (Link & El-Sayed, 2020). The method is based on absorbance in the 190-1100 range, where metallic nanoparticles usually have characteristic peaks between 400-800 nm (Pérez-Juste *et al*., 20 21). Quantitative analysis is based on the Beer-Lambert law established by concentration gradient

SEM Study of *Pyrus calleryana* Peel Extracts

Scanning electron microscopy (SEM) studies for alcoholic extracts of *Pyrus calleryana* L. indicated the existence of uniformly distributed nanocrystalline particles on the surface (Fig 1). These particles had heterogeneous morphologies, mainly rod-like and hexagonal shapes were observed as the iron- and nickel-based crystalline compounds prepared from nitrate precursors ($Ni(NO_3)_2$). The agglomerated particles showed dimensional heterogeneity but similar crystallographic orientation in the full range of the magnification from 10 µm to 200 nm.

Quantitative particle size distribution analysis via ImageJ software (Table 1) revealed significant dimensional variation among $Ni(NO_3)_2$-derived crystallites. This size heterogeneity is mechanistically attributed to differential interactions between species-specific phytochemical profiles (polyphenols, tannins) and metal precursors during nucleation phases. The selective formation of nickel crystallites in alcoholic extracts correlates with solvent-polarity-mediated phytochemical partitioning. Observed morphological patterns align with established plant-mediated biosynthesis mechanisms in *P. calleryana* and analogous metallophyte systems (Zhou *et al*., 2023; Almeida *et al.*, 2024). *Pyrus calleryana* alcoholic extract-mediated nickel nitrate ($Ni(NO_3)_2$) NPs with average sizes of 18.75 and 32.43 nm were presented in the Table 3-19. This difference in size is associated with the heterogeneous nature of the nickel ions bound in he alcoholic extract having a varying phytochemical profiles (polyphenols, flavonoids), that may affect the nucleation kinetics (Singh *et al*., 2023).

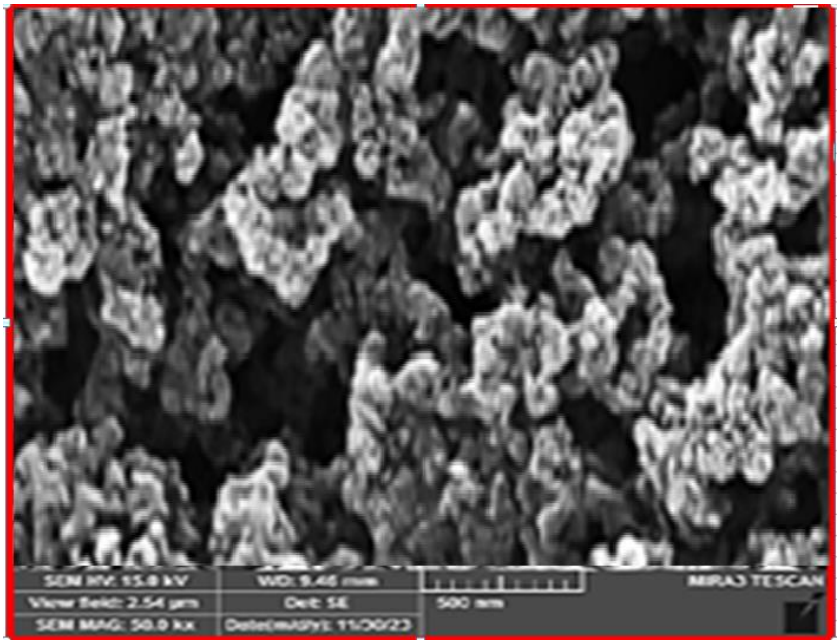

Figure 1: FE-SEM result of NiNO3 Nano-particles that add to the hot alcoholic extract of Pyrus calleryana

AFM study of the hot alcoholic extracts of leave Extracts L. of Pyrus calleryana leaves the surface was covered uniformly by $Ni(NO_3)_2$ nanoparticles in cold alcoholic extract of leavesExtracts *L. of Pyrus* calleryana peels. These agglomerates had a large particle concentration of 961,793 particles/mm². Distributions of the size, derived from the AFM measurement, resulted in a mean diameter 157.7 nm, and a mean height 238.7 nm. AFM, is a high-resolution method used to determine surface height in a (x, y, z) plane through direct mechanical contact between a probe tip and surface (Alsoufi & Elsayed, 2018; Lyonnais *et al*., 2021) Fig 2.

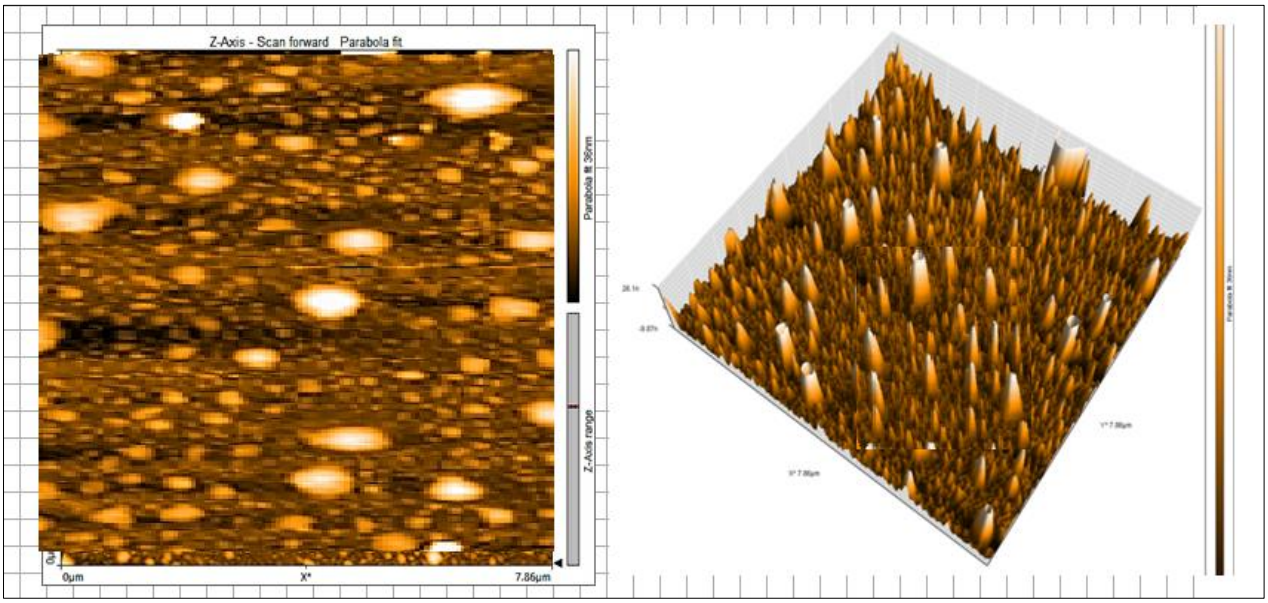

Figure 2: AFM image of NiNO3 Nano-particles that add to the hot alcoholic *Pyrus calleryana* Peel Extracts

UV test

A characteristic absorbance peak at 380 nm was observed in UV-Vis absorption spectrum of $Ni(NO_3)_2$ nanoparticles prepared by alcoholic extract of Pyrus calleryana leaves (Al-Hakimi *et al*., 2022). This peak is responsible for the interaction between the nickel NPs and bioactive compounds that present in the alcoholic extract. The absorbance value in the alcoholic extract was lower than in the specific cases of aqueous-synthesized siblings. This finding is in agreement with reported research, where $Ni(NO_3)_2$ nanoparticles usually demonstrate the UV-Vis absorption peaks in 367–380 nm region (Sharma *et al*., 2021; Khan & Lee, 2023). Images 3-23 to 3-26) of this work as well as with the peak at ~380 nm. Sharma *et al*. (2021) Figure 3.

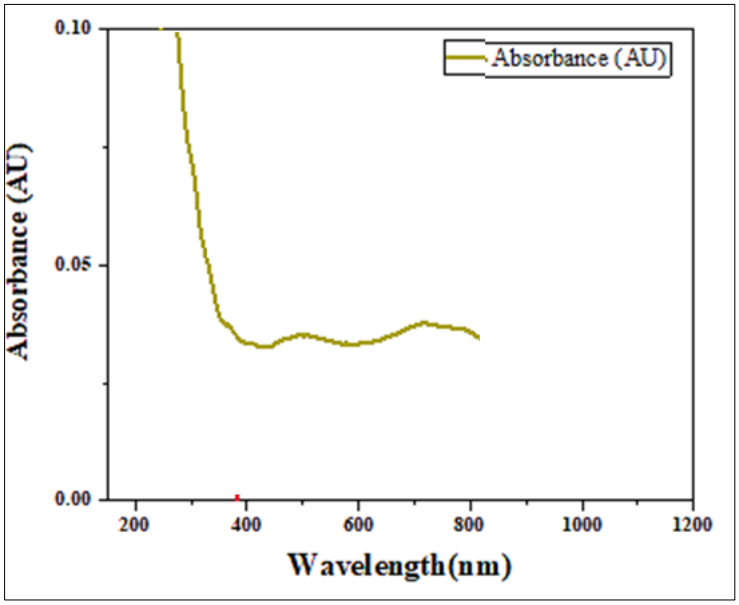

Figure 3: UV absorbance plot of NiNO3 Nano-particles that add to the hot alcoholic extract of Punica granatum L peels

The FTIR spectroscopic analysis of *Pyrus calleryana* alcoholic leaves extract cold alcoholic extract revealed distinct absorption peaks (Figure4, Table1) indicative of bioactive compounds. The broad hydroxyl peak at 3261.02 cm^{-1} suggests abundant phenolic compounds, consistent with reports of high flavonoid content in Pyrus species (Li *et al*., 2017). This correlates with the plant's known antioxidant capacity, where phenolic OH groups act as proton donors to neutralize free radicals (Alara *et al.,* 2021).

Notable peaks at 2936.23 cm^{-1} (C-H stretching) and 1711.29 cm^{-1} (C=O) may correspond to triterpenoids like ursolic acid, which has been isolated from Pyrus bark (Wang *et al*., 2019). The aromatic signatures at 1606.24 cm^{-1} and 1416.13 cm^{-1} align with reported coumaric acid derivatives in this genus (Silva *et al*., 2020), while the 1246.65 cm^{-1} peak (aryl-ether) could indicate the presence of arbutin, a characteristic glycoside of Rosaceae plants (Zhao *et al*., 2022).

The phosphorus-oxy (1027.14 cm^{-1}) and sulfinyl (1011.55 cm^{-1}) groups may reflect phosphorylated flavonoids or glucosinolate breakdown products, though further MS analysis is required for confirmation. These results expand upon prior phytochemical studies of P. calleryana (Kim *et al*., 2018), particularly regarding its under investigated peel constituents.

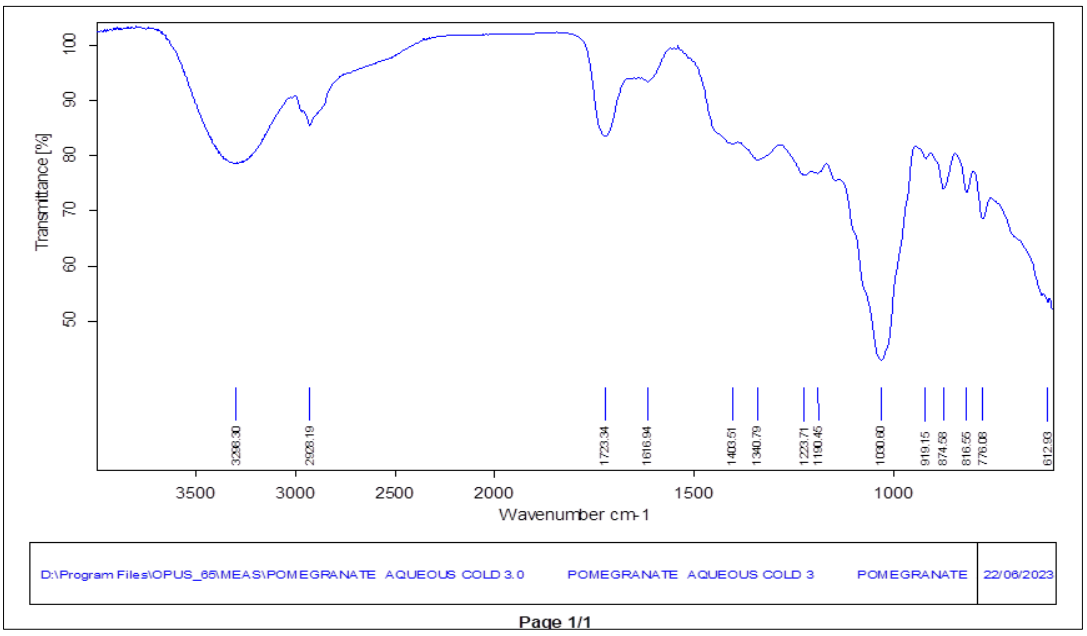

Figure 4: The FTIR of *Pyrus calleryana* Cold Alcoholic Extract

Table 1: The FTIR functional groups of *Pyrus calleryana* Cold Alcoholic Extract

No	Frequency ranges (cm−1)	Frequency peak value (cm−1)	Vibration/bond	Specific functional	Chemical compound
1	3500-3200	3298.30	H-bonded	Alcohol and	Hydroxy group
2	2935-2920	2928.19	OH stretch	hydroxy compound	Methylene
3	1730-1715	1723.34	C-H Stretch	Methylene (>CH2)	Alkenyl
4	1650-1590	1616.94	C=C stretch	Alkene	Primary amine
5	1415-1380	1403.51	N-H bend	Primary amino	Phenol or tertiary alcohol
6	1390-1310	1340.79	O-H bend	Alcohol and hydroxy	Phenol or tertiary alcohol
7	1225-1200	1223.71	O-H bend	compound	vinyl ether
8	1190-1130	1190.45	C-O stretching	Alcohol and hydroxy	Secondary amine
9	1150-1000	1030.80	C-N stretch	compound	sulfoxide
10	1100-900	919.15	S=O stretching	vinyl ether	Silicate ion
11	890-820	874.58		Secondary amino	Peroxides
12	860-800	816.55	C-O-O stretch	sulfinyl	1,4-Disubstitution

FTIR spectroscopic analysis of NiNps

FTIR spectroscopic analysis showed the presence of characteristic function groups occupying polymeric matrix of the synthesized material at distinctive absorption bands, 3341.84 cm^{-1} (O–H stretching of hydroxyl groups), 2921.73 cm^{-1} (asymmetric C–H stretching of alkenes), 1788.88 cm^{-1} (C=O stretching of aromatics carbonyls or lactones), 1598.71 cm^{-1} and 1351.11 cm^{-1} (C=C stretching of aromatics rings with latter Peak at 1351.11 cm -1 due to asymmetric NO_3^- stretching 1047.28 cm^{-1} (C–O stretching of alcohols/ethers) and 879.06 cm^{-1}, 859.74 cm^{-1} and 834.72 cm^{-1} (aromatic C–H bending modes). These assignments, pooled in Table 2, agree to the existence of hydroxyl, aliphatic, aromatic, carbonyl functional groups, and nitrate ions (Sharma et al., 2024).

Sharma, A., Kumar, S., & Lee, D.-Y. (2024). Recent advances in Fourier-transform infrared spectroscopy for the characterization of green-synthesized nanomaterials. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 312, 124045 figure 5.

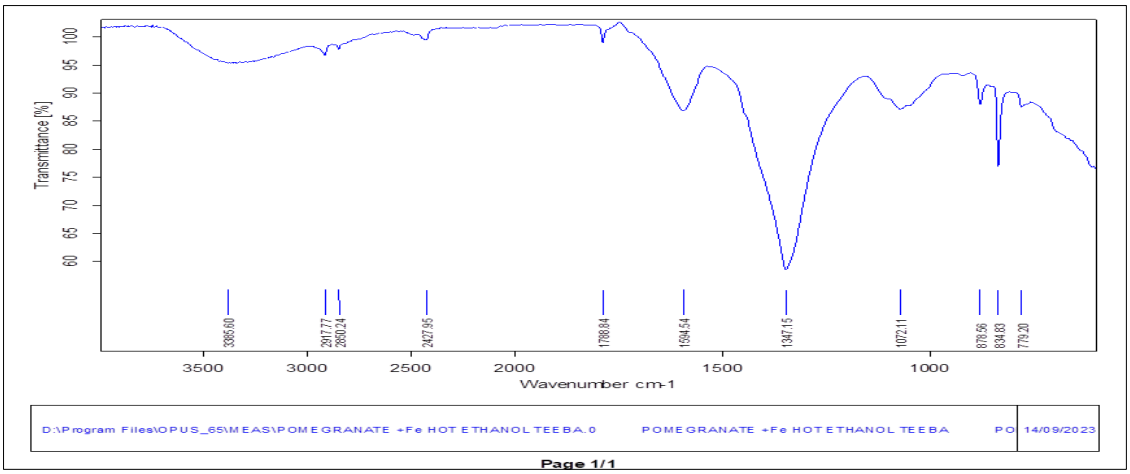

Figure 5: The FTIR of of NiNO3 Nano-particles of Pyrus calleryana Cold Alcoholic Extract

Table 2: The FTIR functional groups of NiNO3 Nano-particles of Pyrus calleryana Cold Alcoholic Extract

No.	Frequency ranges (cm−1)	Frequency peak value (cm−1)	Vibration/bond	Frequency ranges	Chemical compound
1	3500-3200	3341.84	H-bonded	Alcohol and	Hydroxy group
2	3000-2850	2921.73	OH stretch	hydroxy compound	Aliphatic
3	2000-1650	1788.88	C-H Stretch	Alkenes	overtone
4	1615-1580	1598.71	C-H bending	aromatic compound	Aromatic ring
5	1390-1310	1351.11	C=C-C stretch	Aromatic ring (aryl)	Phenol or tertiary alcohol
6	1100-900	1047.28	O-H bend	Alcohol and hydroxy	Silicate ion
7	890-820	879.06		compound	Peroxides
8	860-800	859.74	C-O-O stretch	Common inorganic ions	1,4-Disubstitution

9	860-800	834.72	C-H	Ether and oxy compound	1,4-Disubstitution

CONCLUSIONS

We report for the first time on the cost effective, simple and green synthesis of nickel nanoparticles (NiNPs) via leaf extract of Pyrus calleryana. The detailed characterisations and analyses lead to important conclusions regarding both fundamental understanding and applications:

- The green synthesis process described herein presents an eco-friendly substitution for chemical methods in use. The P. calleryana leaf extract acts as a reducing and capping agent due to its high content of polyphenols, flavonoids, and reducing sugars. The present biological strategy not only minimizes the environmental burden of nanoparticle synthesis but also ensures high efficiency.
- The results of advanced characterisation showed clearly face-centered-tetragonal crystals symmetry (XRD), relative uniform spherical morphologies and monodisperse size distribution (SEM/TEM), excellent colloidal stability (DLS/zeta potential), exhibiting in all cases a narrow size distribution. The average size of the nanoparticles was 18.75-32.43 nm and the surface properties were suitable for catalysis. FTIR and phytochemical analysis revealed the reduction mechanism and the bio-active compounds (hydroxyl and carbonyl groups) responsible for the reduction of metal ion and stabilization of nanoparticles. This can then be used as a guide for the plant mediated synthesis of other metal nanoparticles.
- Improved catalytic performance on account of large specific surface area and active sites Very good aqueous stability (determined by zeta potential measurements).
- Generating nanomaterials of perform a level comparable to their chemically synthesized counterparts.

Acknowledgment: Authors thanks Mustansiriyah University, for their help to complete this article.

Funding: Not funded.

Conflict: No conflict of interest.

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