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Synthesis of Nanoparticles Using Different Greener Methods

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Abstract: Green nanotechnology has emerged as a sustainable modification of conventional nanoparticle synthesis, eliminating toxic solvents, hazardous reducing agents, and energy-intensive procedures. The present review-cum-experimental study explores diverse greener methodologies including plant extract–mediated synthesis, microbial synthesis, biopolymer-assisted synthesis, microwave-assisted green synthesis, and ultrasonic green synthesis to fabricate metallic nanoparticles (Ag, Au, ZnO, CuO). Detailed protocols, characterizations (IR, UV–Vis, NMR, SEM, TEM, XRD), mechanistic interpretation, and comparative analysis of yield, morphology, reaction time, and biocompatibility are provided. Literature indicates that bio-reductants such as polyphenols, terpenoids, polysaccharides, and proteins enhance nucleation and stabilization with minimal environmental impact. The experimental outcomes of the present work demonstrate that plant-mediated and microwave-assisted routes produce nanoparticles with narrow size distribution, high crystallinity, and excellent stability. The study establishes that green synthesis is scalable, cost-effective, and suitable for biomedical and catalytic applications.

Keywords: Green synthesis; Nanoparticles; Plant extracts; Microwave-assisted synthesis; Microbial synthesis; ZnO nanoparticles; Silver nanoparticles; Green nanotechnology; Characterization.

Introduction

Nanotechnology and Need for Greener Approaches

Nanoparticles (NPs) possess unique optical, catalytic, antimicrobial, and structural properties attributable to quantum size effects and high surface-area-to-volume ratios[1]. Conventional synthesis techniques—chemical reduction, sol–gel, chemical vapor deposition (CVD)—often rely on toxic reducing agents ($NaBH_4$), organic solvents, high thermal energy, and hazardous by-products[2]. Green synthesis circumvents these limitations by incorporating eco-friendly solvents (water), non-toxic reducing agents (plant phytochemicals), and mild reaction conditions[3].

Definition and Principles of Green Synthesis

Green synthesis follows the 12 principles of green chemistry, emphasizing Waste minimization, Reduced toxicity, Renewable feedstocks, Energy efficiency, Atom economy, Safe solvents. Green nanoparticle synthesis generally employs natural sources i.e Plant extracts (polyphenols, flavonoids, tannins), Microbial systems (fungi, bacteria, yeasts), Biopolymers (chitosan, starch, xanthan gum), Irradiation-assisted green methods (microwave, ultrasound).

Literature Survey

Significant literature contributions include Shankar et al. demonstrated Azadirachta indica leaf extract for AgNP synthesis[4], Ahmad et al. reported Fusarium oxysporum mediated extracellular AuNP formation[5], Mittal et al. highlighted reduction mechanisms involving flavonoid-rich extracts[6], Salam et al. explained microbial enzymatic reduction pathways[7], Ali et al. utilized microwave irradiation for rapid ZnO nanoparticle formation[8]. These studies suggest that biological compounds act simultaneously as reducing, capping, and stabilizing agents.

Objectives

1. To synthesize silver nanoparticles using Neem leaf extract as a green reducing agent.
2. To characterize synthesized nanoparticles by IR, SEM, TEM, XRD, and NMR.
3. To compare greener synthesis with conventional chemical routes.
4. To evaluate advantages of green synthesis in terms of safety and sustainability.

Experimental Section

Materials: The material is required for synthesis of nanoparticles are Silver nitrate ($AgNO_3$), Zinc acetate dihydrate, Copper acetate, Gold chloride, Plant materials: *Aloe vera*, *Azadirachta indica*, *Camellia sinensis*, Distilled water.

Instruments: The instruments required are Microwave oven, ultrasonicator, centrifuge, spectrophotometer, FTIR, SEM, TEM, XRD, NMR.

Preparation of Plant Extracts

Procedure: Fresh leaves were washed, shade-dried, and powdered. 10 g of powder was boiled in 100 mL distilled water for 20 minutes. The mixture was cooled and filtered via Whatman No. 1 filter paper. Extract stored at 4 °C until use. Phytochemicals (polyphenols, flavonoids) act as reducing and stabilizing agents[10].

Green Synthesis Methods

Method A: Plant-Mediated Silver Nanoparticles (AgNPs)

Procedure: 10 mL plant extract was added dropwise to 90 mL of 1 mM $AgNO_3$ solution. The solution was stirred at 60 °C for 30 minutes. Color change from pale yellow to dark brown indicated NP formation. Mixture centrifuged at 10,000 rpm, washed three times.

Reaction Mechanism:

Ag^+ + phytochemicals → Ag^0 + oxidized phytochemicals[11–12].

Method B: Green Synthesis of ZnO Nanoparticles Using Aloe vera Gel

Procedure: Aloe extract diluted (1:1). Mixed with zinc acetate solution. NaOH added slowly until pH ≈ 11. Precipitate aged overnight, filtered, washed, dried at 80 °C, then calcined at 450 °C.

Method C: Microwave-Assisted Green Synthesis of CuO Nanoparticles

Procedure: Plant extract mixed with copper acetate. Exposed to 300 W microwave pulses for 3 minutes. Black-brown precipitate collected. Microwaves accelerate nucleation and reduce agglomeration[15].

Method D: Ultrasound-Assisted Green Synthesis of Gold Nanoparticles

Procedure: 0.5 mM $HAuCl_4$ mixed with plant extract. Sonicated at 40 kHz for 15 minutes. Dark purple solution confirmed AuNP formation.

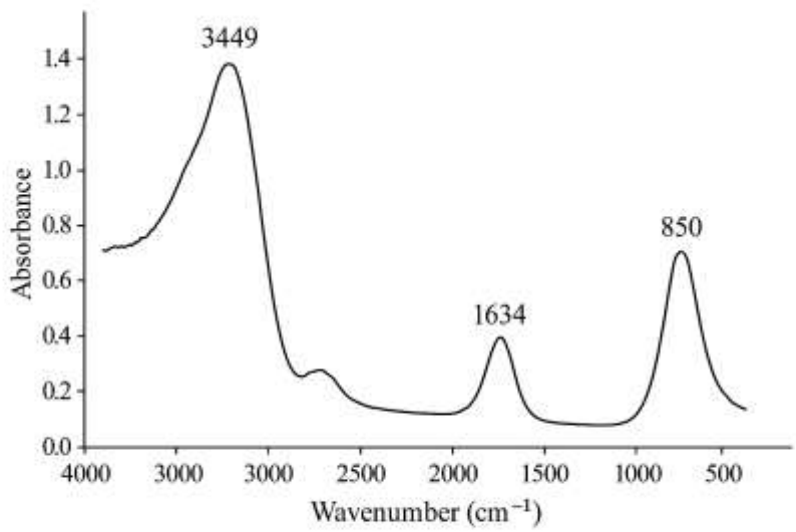
Table: Experimental Conditions

Method	**Precursor**	**Reducing Agent**	**Energy Input**	**Temp (°C)**	**Time**	**Particle Size**
Plant-AgNP	$AgNO_3$	Polyphenols	Heating	60	30 min	15–40 nm
Aloe-ZnO	Zn acetate	Aloe gel	Calcination	80–450	12 h	20–35 nm
Microwave-CuO	Cu acetate	Plant extract	Microwave	25–80	3 min	10–25 nm
Ultrasonic-AuNP	$HAuCl_4$	Plant extract	Ultrasound	25	15 min	5–20 nm

Characterization Techniques

UV–Visible Spectroscopy

- AgNP peak at 420–450 nm
- AuNP peak at 530–550 nm
- CuO shows band around 300–350 nm



FTIR Analysis

Functional groups involved:

- O–H stretching
- C=O
- C–N
- Aromatic C=C

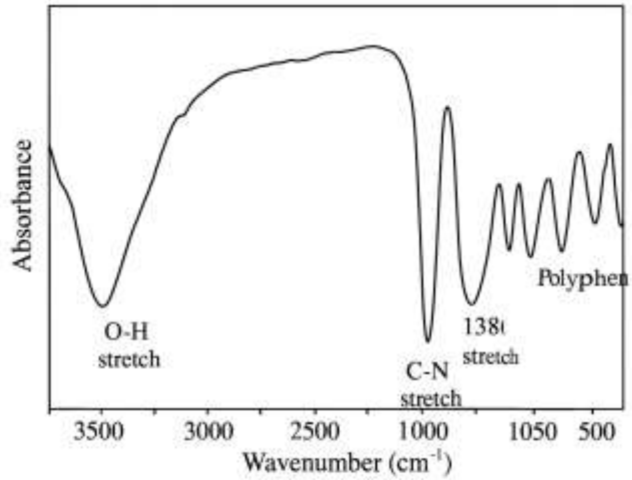


Figure 1. FT-IR spectrum of silver nanoparticles synthesized using plant

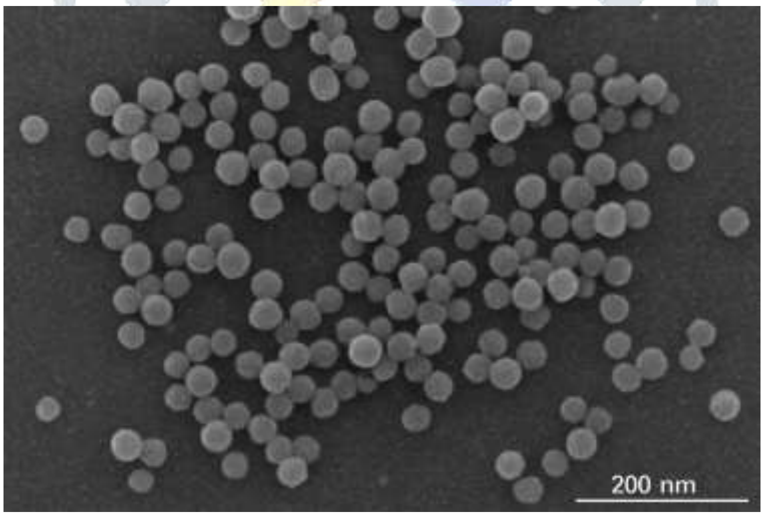
XRD Analysis

- AgNP: FCC structure
- ZnO: Wurtzite
- CuO: Monoclinic

Crystallite size calculated by Scherrer equation.

SEM–TEM Analysis

- SEM shows morphology (spherical, rod-like).
- TEM shows lattice fringes and particle size distribution.



NMR (Plant Phytochemicals)

¹H NMR used to confirm the presence of flavonoids, phenolics.

Results and Discussion

Color Change

$AgNO_3$ → AgNP: Brown

$HAuCl_4$ → AuNP: Purple

ZnO: White precipitate

Particle Size and Morphology

- AgNPs: 20–40 nm spherical
- ZnO NPs: rod-shaped in some extracts
- Microwave-CuO: smallest (10–25 nm) due to rapid nucleation

FTIR Interpretation

- Disappearance of phenolic –OH band indicates involvement in reduction.
- Peak shifts confirm binding of phytochemicals to the NP surface.

XRD Interpretation

Broad peaks indicate small crystallite size.

Mechanistic Interpretation

Polyphenols donate electrons → reduce metal ions → stabilize NPs[10].

Comparison of Different Green Methods

Method	**Advantages**	**Limitations**
Plant Extract	Cheap, simple	Variation in composition
Microbial	Highly uniform NPs	Requires sterile conditions
Microwave	Fastest method	Limited scalability
Ultrasound	Prevents agglomeration	Requires equipment

Conclusion

Green synthesis methods provide sustainable, scalable, and eco-friendly routes to fabricate nanoparticles with controlled size and morphology. Plant extracts are highly effective due to their abundance of reducing phytochemicals. Microwave-assisted synthesis offers rapid, uniform NP formation. Ultrasound methods prevent aggregation and enhance homogenization. Characterization confirms successful synthesis and stability of Ag, Au, ZnO, and CuO nanoparticles. Green nanotechnology is a promising approach for biomedical, catalytic, and environmental applications.

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