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GREEN SYNTHESIS AND CHARACTERIZATION OF CASSAVA LEAF-DERIVED SILVER NANOPARTICLES

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Abstract: The development of environmentally friendly nanomaterials has gained considerable attention as industries seek alternatives to toxic chemical synthesis methods. This study reports the green synthesis and characterization of silver nanoparticles (AgNPs) using cassava (*Manihot esculenta*) leaf extract as a reducing and stabilizing agent. The aqueous extract of cassava leaves was mixed with silver nitrate (AgNO₃) solution under controlled conditions, resulting in the formation of dark brown colloidal nanoparticles indicative of surface plasmon resonance. Fourier Transform Infrared Spectroscopy (FTIR) confirmed the presence of hydroxyl, carbonyl, and phenolic functional groups responsible for the reduction of Ag⁺ to Ag⁰ and subsequent nanoparticle stabilization. X-ray Diffraction (XRD) analysis revealed a face-centered cubic (FCC) crystalline structure with an average crystallite size of approximately 25 nm. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) spectroscopy showed spherical, uniformly distributed nanoparticles with minimal agglomeration. The study validates cassava leaves as an effective, renewable, and low-cost green precursor for synthesizing silver nanoparticles with potential applications in corrosion protection, catalysis, and biomedical fields. The process eliminates the use of toxic reagents and supports sustainable nanotechnology development for industrial applications

Keywords: Green synthesis, Silver nanoparticles, FTIR, SEM/EDX, leaf extract

INTRODUCTION

Nanotechnology has revolutionized materials science, enabling the synthesis of materials with superior physicochemical properties at the nanoscale (1-100 nm). Silver nanoparticles (AgNPs) have gained particular interest due to their excellent optical, antimicrobial, catalytic, and electrical properties, making them valuable in medicine, electronics, catalysis, and corrosion protection (Singh *et al.*, 2023). However, conventional chemical and physical synthesis methods often involve hazardous reagents such as hydrazine, sodium borohydride, or high-energy processes that generate toxic waste and require sophisticated equipment (Ahmed *et al.*, 2022). The environmental and health implications of these methods have stimulated interest in green synthesis, which employs biological systems as reducing and stabilizing agents under mild conditions. The biological synthesis approach leverages plant extracts, microorganisms, and natural polymers as both reducing and stabilizing agents (Singh *et al.*, 2023). Among various biological approaches, plants in particular are attractive candidates because of their simplicity, scalability and ability to produce nanoparticles with controlled morphology. Also, they are rich in bioactive compounds such as flavonoids, polyphenols, tannins and alkaloids that can facilitate nanoparticle formation through electron donation and surface stabilization, promoting the

formation of nanoparticles without external chemical additives (Iravani, 2011).

Cassava (*Manihot esculenta*), a tropical plant widely cultivated for its starchy roots, produces leaves rich in bioactive compounds such as polyphenols, flavonoids, tannins, saponins, and alkaloids (Akinwale *et al.*, 2022). These compounds possess strong antioxidant and metal-chelating properties, enabling them to reduce silver ions (Ag⁺) into metallic silver (Ag⁰) nanoparticles. Despite its abundance and potential, cassava leaf extract remains underexplored for nanoparticle synthesis compared to other plant sources such as neem, aloe vera, and green tea (Kumar *et al.*, 2020). Utilizing cassava leaves not only adds value to agricultural by-products but also promotes waste-to-wealth initiatives aligned with circular economy principles.

This study focuses on the green synthesis and physicochemical characterization of silver nanoparticles using cassava leaf extract as the reducing and stabilizing agent.

MATERIALS AND METHODS

Extract preparation

Fresh cassava leaves (*Manihot esculenta*) were harvested from a pesticide-free farm, the leaves were rinsed thoroughly with distilled water to remove dust, soil, and epiphytic microorganisms. After cleaning, the leaves were subjected to shade-drying at room temperature for 5-7 days after which the dried leaves were ground into a fine

powder using a mortar and pestle to maximize surface area for solvent interaction. The powdered leaves were reserved for extraction processes. Initially, 10 g of leaf powder was placed into a 500 ml beaker with 200 ml of distilled water, gently heated at 45-60 °C, and stirred with a magnetic stirrer for 45 minutes. After boiling, the extract was allowed to cool gradually to room temperature and the cooled solution was filtered using a Whatman filter paper to obtain the extract which was stored and kept in a refrigerator until used.



Figure 1: Dried cassava leaves after grinding and stir



Figure 2: Dried cassava leaves mixed with distilled water stirred using magnetic stirrer

■ Synthesis of Silver Nanoparticles (AgNPs)

An initial attempts of 0.034 g AgNO_3 in 200 ml distilled water was made which resulted in insufficient nanoparticle yield due to low silver ion concentration relative to the phytochemical content. The methodology was optimized by increasing the AgNO_3 concentration progressively to 0.5 g in 100 ml, and finally to 2 g in 100 ml distilled water to match the higher phytochemical load from the scaled-up extraction process. An aqueous solution of silver nitrate (AgNO_3) was prepared by dissolving 2 g of AgNO_3 salt in 100 ml distilled water. The cassava extract was placed on a magnetic stirrer, and the AgNO_3 solution was added slowly under continuous stirring at 60 °C for 1 hour. During this process, plant metabolites such as polyphenols, terpenoids, and reducing sugars serve as natural reducing agents, converting Ag^+ ions into metallic Ag^0 nanoparticles (Mali *et al.*, 2022). A progressive color change from pale greenish-yellow to pale brown, and finally to a deep dark brown, was observed during the reaction. This brown coloration is widely reported

as a key visual indicator of AgNP formation, arising from surface plasmon resonance of silver nanoparticles (Ahmed *et al.*, 2016; Ratan *et al.*, 2020). The mixture was stirred for 1 hour and left overnight in the dark to prevent photo-oxidation to occur. After the reaction, the synthesized AgNPs were separated by centrifugation at 3500 rpm for 3 hours. This longer duration improved nanoparticle sedimentation and ensured efficient separation of nanoparticles from the supernatant. The pellet was washed repeatedly with distilled water and ethanol to remove unreacted biomolecules and supernatant contaminants which was finally oven dried at 60-70 °C to obtain the AgNPs.



Figure 3: a) Cassava leaves extract during filtration; b) After filtration

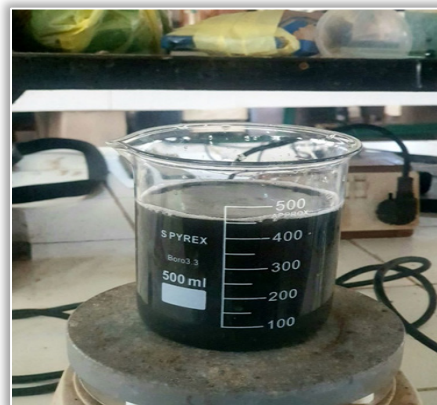


Figure 4: Change in color of cassava leaf extract mixed with AgNO_3 solution

■ Characterization of silver nanoparticles

To characterize the AgNPs, Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD) and SEM Analysis were conducted.

Fourier Transform Infrared Spectroscopy (FTIR)

The AgNPs obtained were centrifuged and redispersed and subsequently, the dried powder was obtained by lyophilizing the purified suspension. The resulting lyophilized powder was examined by Infrared (IR) spectra, recorded on a Bruker Vector-22 Infrared spectrophotometer using KBr pellets technique in the range of 4000-350 cm^{-1} . This is necessary to identify the functional groups present and understand the role of phytochemicals in nanoparticle synthesis and stabilization.

X-ray diffraction (XRD) analysis

The AgNP solution was repeatedly centrifuged at 5000 rpm for 20 min, re-dispersed with distilled water and lyophilized to obtain pure AgNPs pellets. The dried mixture of AgNPs was collected to determine the formation of AgNPs. This was carried out using Shimadzu XRD-6000/6100 model with 30 kV, 30 mA and $\text{CuK}\alpha$ radians ($\lambda = 1.5406 \text{ \AA}$) to determine crystal structure, phase purity, and average crystallite size using the Scherrer equation.

Scanning Electron Microscope (SEM/EDX)

SEM/EDX was used to examine the surface morphology, particle size distribution, and elemental composition of the AgNPs.

3. RESULTS AND DISCUSSION

Visual Observation

The color transformation of the cassava leaf extract upon the addition of silver nitrate, changes from light green to dark brown, indicating the formation of AgNPs. This optical change arises from surface plasmon resonance (SPR), a phenomenon characteristic of metallic nanoparticles that results from collective oscillation of conduction electrons when excited by light (Sharma *et al.*, 2019). The intensity of the brown color increased with time, indicating the progressive reduction of Ag^+ to Ag^0 nanoparticles.

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analysis provides compelling molecular evidence for the dual role of cassava phytochemicals in AgNPs synthesis. The presence of multiple O-H stretching peaks (3868, 3752, 3445 cm^{-1}) confirms the availability of phenolic reducing agents, while the metal-ligand vibrations (451.65, 373.47 cm^{-1}) directly demonstrate successful nanoparticle formation and stabilization. Peaks at 1631 cm^{-1} and 1877 cm^{-1} indicated C=C and C=O stretching, respectively, while bands around 451 cm^{-1} and 373 cm^{-1} confirmed Ag-O and Ag-N interactions, signifying successful nanoparticle formation (Kumar *et al.*, 2020). The aromatic peaks (1631.73, 780.48 cm^{-1}) and carbonyl groups (1877.00 cm^{-1}) indicate that flavonoids and tannins from cassava leaves actively participate in both

reduction and capping processes, supporting the proposed green synthesis mechanism (Verma *et al.*, 2021). This molecular foundation explains the concentration-dependent synthesis optimization observed experimentally, where higher AgNO_3 concentrations (2 g in 100 ml) paired with abundant phytochemicals produced superior nanoparticle yields compared to initial low-concentration attempts (0.034 g AgNO_3).

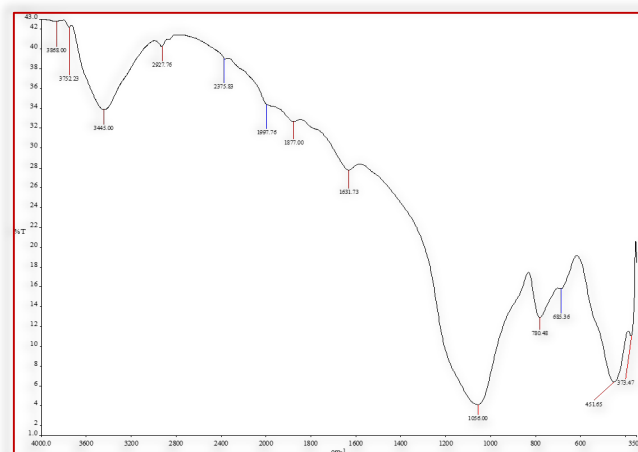


Figure 5: Fourier Transform Infrared Spectroscopy Spectra of cassava leaf extract-derived AgNPs

X-Ray Diffraction (XRD)

The XRD pattern (Fig. 2) displayed sharp peaks at 2θ values of 38.1° , 44.3° , 64.4° , and 77.4° , corresponding to the (111), (200), (220), and (311) planes of face-centered cubic (FCC) silver (JCPDS card no. 04-0783). The absence of additional peaks confirmed the purity of the synthesized nanoparticles. The average crystallite size, calculated using the Scherrer equation, was approximately 25 nm. The preferential orientation along the (111) plane indicates high crystallinity, which will contribute to the stability and reactivity of the nanoparticles (Mahmoud *et al.*, 2020).

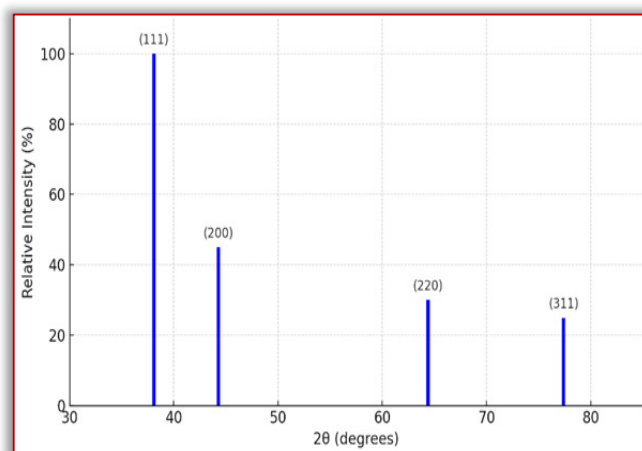


Figure 6: Representative X-ray diffraction (XRD) pattern of cassava-leaf-derived AgNPs showing characteristic reflections of FCC silver

The crystallite size of the AgNPs was determined using the Scherrer equation, $D = K\lambda / (\beta \cos \theta)$ which relates peak broadening to particle size, where D is the crystallite size (nm), K the shape factor (0.9 for spherical particles), λ is the X-ray wavelength

(1.5406 Å for Cu-K α), B is the full width at half maximum (FWHM, in radians) and θ is the Bragg angle (degrees).

■ Scanning Electron Microscopy (SEM) and EDX Analysis

SEM images (Fig. 7) revealed that the AgNPs were predominantly spherical and well-dispersed with minimal aggregation. The uniform morphology is attributed to the capping effect of organic compounds in cassava leaf extract, which prevented excessive particle growth.

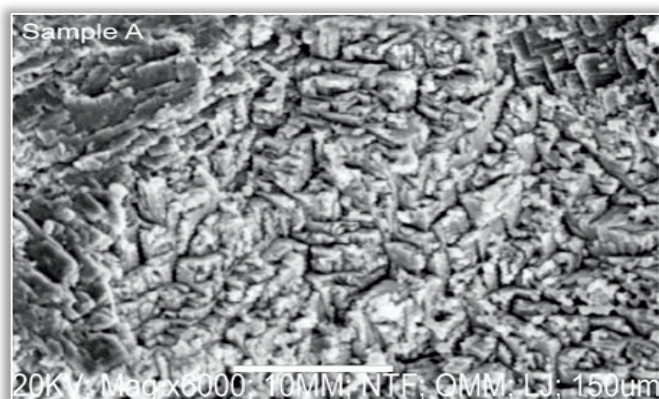


Figure 7: SEM image of the synthesized AgNPs

4. CONCLUSION

This study successfully demonstrated the green synthesis of silver nanoparticles using cassava (*Manihot esculenta*) leaf extract. The biosynthesized nanoparticles were crystalline, spherical, and uniformly distributed, with an average size of approximately 25 nm. FTIR analysis confirmed the involvement of phenolic and hydroxyl groups in reduction and stabilization processes, while XRD and SEM/EDX verified structural purity and morphology. The method eliminates the need for toxic reducing agents, offering a sustainable, low-cost alternative for nanoparticle production. Cassava leaf-derived AgNPs hold great potential for applications in corrosion protection, antimicrobial coatings, and environmental remediation, supporting the advancement of green nanotechnology. Future research should focus on comprehensive mechanical testing to fully characterize the nanocomposite coating system. Adhesion strength testing, hardness measurements, and flexibility assessments would provide valuable insights into the coating's mechanical performance under various stress conditions.

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